Exhibit 110

IN THE UNITED STATES DISTRICT COURT FOR THE DISTRICT OF NEW JERSEY

IN RE: JOHNSON & JOHNSON TALCUM POWDER PRODUCTS MARKETING, SALES PRACTICES AND PRODUCTS LIABILITY LITIGATION

THIS DOCUMENT RELATES TO ALL CASES

MDL NO. 16-2738 (FLW) (LHG)

EXPERT REPORT OF M. DARBY DYAR, PHD FOR GENERAL CAUSATION DAUBERT HEARING

Date: February 25, 2019

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I. EXECUTIVE SUMMARY

To confirm the presence of asbestos in talcum powder, there are three questions to consider:

- **1. Mineralogy**: Are the mineral species present among those that include asbestos?
- **2. Physical properties (habit)**: Do the minerals have an "asbestiform" habit, occurring as particles that are lengthwise separable into flexible fibers?
- **3. Concentration**: What are the abundances of the minerals?

The expert reports dated November 14, 2018, January 15, 2019 and February 2, 2019 from plaintiffs' experts William E. Longo and Mark W. Rigler (the "Longo/Rigler MDL Reports") fail to demonstrate that **any** of these three requirements is true of the impurities allegedly occurring in the tested talcum powder. Instead, they (or more specifically, their five analysts) applied a methodology that was inherently designed to achieve the results they desired for purposes of this litigation.

As an initial matter, many minerals have very similar chemical formulas, and many minerals occur with fibrous shapes, but not all of these minerals are "asbestos." Asbestos minerals have both a specific chemical composition and a unique 'habit' or morphology. Asbestos is defined as one of six particular minerals exhibiting the characteristics of an asbestiform habit, meaning they can be separated into flexible fibers with high tensile strength. Drs. Longo and Rigler confuse "counting criteria" with this definition, using a disingenuous distinction between what they call the "geological definition" and what they wrongly invent as the "regulatory definition." This is not scientifically proper.

- EDS (mineral chemistry). Drs. Longo and Rigler opine on tests run by their analysts using Energy Dispersive Spectroscopy ("EDS") to identify the chemistry of certain particles that they have isolated in the samples tested. EDS, properly used, can somewhat measure the chemistry of a mineral, or at least narrow down the possible chemistries, even if it cannot tell a scientist whether that mineral's shape is asbestiform or not. But the analysts do not follow generally accepted scientific methodology in their EDS analysis. For example:
 - o They "eyeball" the EDS spectra and rely only on the images generated by an EDS analysis to evaluate the chemical composition of each mineral.
 - o They visually compare their spectra to "reference standards" apparently belonging to their lab only, which standards are not in the published literature, which likely represent only asbestiform minerals and which Drs. Longo and Rigler did not produce (such that it cannot be determined if these standards have any scientific validity whatsoever).
 - o In doing so, they deliberately choose not to generate **quantitative numbers** that would more accurately determine chemical compositions, which is the very purpose of an EDS analysis of an unknown mineral. The quantitative data is necessary to make scientifically valid conclusions, and their conclusions are therefore unsupportable.

As a result, Drs. Longo and Rigler and their analysts used a flawed methodology that resulted in wildly inconsistent results across different analysts and over time. In science, proof involves demonstrating that a result is replicable. Consequently, the particles that they identify as "anthophyllite" may actually be talc, cummingtonite or other minerals. The particles that they identify as "tremolite" could be other amphiboles¹, diopside (pyroxene) or other minerals.

Finally, confirming the lack of reproducibility in their methodologies, I have analyzed the EDS interpretations of each of the five separate analysts used by Drs. Longo and Rigler. Although there should be parallel results across analysts, there is clear evidence of personal biases, which is unsurprising given the unscientific and subjective nature of the methodologies employed.

- **SAED** (**crystal structure**). Drs. Longo and Rigler opine on tests run by their analysts using selected area electron diffraction (SAED) to identify the crystal structure of selected particles in the samples tested. Again, they do not follow generally accepted scientific methodology in their SAED analysis. For example:
 - o A majority of the patterns they generate are of poor quality, making them impractical or impossible to interpret.
 - o For an accurate identification of an unknown mineral, at least two (and preferably more) SAED patterns must be taken with the sample oriented along a "zone axis." Yet for those minerals identified as "tremolite," they use only one SAED pattern, resulting in inconclusive data that would be consistent with many other minerals, including those that have no asbestiform habit. Similarly, for those minerals that they identify as "anthophyllite," the analysts took two SAED patterns for only two samples, and these are insufficient to uniquely identify anthophyllite.
 - O A fundamental part of analyzing SAED results is to calculate the "d-spacings" between layers of atoms, which may be diagnostic of each mineral species. It appears that Drs. Longo and Rigler and their analysts did not initially use d-spacings at all, once again adopting qualitative "eyeball" methodologies found nowhere in the literature over quantitative, accepted science. **After** generating their first report, they used a small number of d-spacing measurements to "verify" their prior conclusions. Yet those "verifications" show d-spacings that are consistent with 25% of all common rockforming minerals. Even Drs. Longo and Rigler concede they do not uniquely identify any one mineral.
 - o Some of the Longo/Rigler *d*-spacing interpretations even have impossible values, demonstrating significant flaws in the methodology used.

¹ Amphibole is the group name of a group of hydrous silicate minerals that include, among other individual minerals, tremolite, actinolite and anthophyllite.

² The zone axis is the direct crystal axis that is parallel to the electron beam at the orientation when the SAED pattern is recorded

o The EPA "Yamate III" protocol for analyzing talc for impurities – which Drs. Longo and Rigler say they followed – requires two SAED zone axis determinations. Yet, of the more than 70 bottles tested, Drs. Longo and Rigler only did this for **two** bottles.

As a result, what Drs. Longo and Rigler identify as anthophyllite or tremolite in the samples analyzed may actually be hundreds of other possible minerals or individual crystal structures.

- **TEM visual inspection**. The analysts in the Longo/Rigler MDL Reports purport to find the concentration of "asbestos" using visual inspection techniques under TEM. They do not, however, follow generally accepted scientific methodology in this analysis. For example:
 - Use of TEM images to **visually** distinguish non-asbestiform amphiboles and asbestiform amphiboles is scientifically invalid, as stated by the very protocols upon which Drs. Longo and Rigler rely.
 - Indeed, Drs. Longo and Rigler and their analysts did not apply objective criteria for distinguishing between cleavage fragments, bundles and fibers, as demonstrated by the inconsistent results of their analysts.
 - Moreover, the results reported in the Longo/Rigler MDL Reports are inconsistent from analyst to analyst, highlighting the unreliability of their method of visual identification using TEM.
 - O The analysts used by Drs. Longo and Rigler utilized the concept of "aspect ratio" the ratio of a particle's length to width and counted all particles greater than 0.5 μm in length that have substantially parallel sides and an aspect ratio greater than 5:1. Yet, as Drs. Longo and Rigler admit, a particle that is non-asbestiform can also have an aspect ratio greater than 5:1. This means that the Longo/Rigler MDL Reports do not distinguish asbestiform minerals from non-asbestiform minerals (e.g., "cleavage fragments") in this regard.
 - This is scientifically invalid because, based on peer-reviewed studies, it is known that size distributions for non-asbestiform particles are different than those for asbestiform particles. Using statistical methods methods not used by Drs. Longo and Rigler asbestiform and non-asbestiform populations can readily be distinguished. However, Drs. Longo and Rigler deliberately did not compile data in the low aspect ratio range, making a valid particle analysis impossible.
 - To the extent analysis is possible of the aspect ratio size population they did measure, statistical tests of aspect ratio populations establish that the particles measured by the analysts used by Drs. Longo and Rigler belong to the population represented by nonasbestiform tremolite.
- **PLM** (mineral identification). The analysts used polarizing light microscope (PLM) images to identify the nature of any asbestos in the tested samples and the concentration. They did not, however, follow generally accepted scientific methodology in their PLM analysis. For example:

- o The analysts purported to follow the methodology found in a peer-reviewed article by Su (2003). But Su sets forth a particular methodology to do dispersion staining, an absolutely critical step of the analysis, which was not followed.
- Otherwise, the PLM images from the Longo/Rigler MDL Reports are in many cases ambiguous because talc and amphiboles have overlapping and similar optical properties, and they can easily be confused for each other.
- o The analysts doing the PLM work purported to use two methodologies to determine concentration of asbestos contaminants "ISO" PLM and "Blount" PLM which should have consistent results. All tests using the former were either negative or under 0.1% by weight percentage (consistent with the absence of asbestos). Ten of the tests using the latter were positive at concentrations up to seven times the results of the former, which indicates a complete lack of reliability and reproducibility in this PLM testing.
- o Dr. Longo and Rigler's "concentration" analysis did not use "point counting," which is the standard method to estimate concentration of phases by PLM. Rather, Drs. Longo and Rigler's analysts used, once again, visual reference charts that appear to be unique to their lab, are not in the peer-reviewed literature, and are not produced (such that it cannot be determined if these standards have any scientific validity whatsoever).

In summary, the results in the Longo/Rigler MDL Reports are the product of fundamentally flawed methodologies and are **not the result of work done according to generally accepted scientific standards and methodologies.** Despite producing lengthy, descriptive reports and undertaking extensive measurements according to government protocols, Drs. Longo and Rigler did not properly train their personnel for the task of characterizing impurities in talc, where the possible mineralogy is unconstrained. Thus, they lack the background to understand or interpret their results, especially where, as here, the impurities are tiny, and many possible minerals can be present.

The Longo/Rigler MDL Reports simply fail to demonstrate that *any* asbestos minerals are present in the material Drs. Longo and Rigler tested in *any* concentration with *any* asbestiform shapes.³ Indeed, every methodological error in their testing results in false positive findings of asbestos, but not false negatives. Given that Drs. Longo and Rigler's testing can only be described as a series of errors, each of which acts to amplify the errors preceding it, the results of Drs. Longo and Rigler's testing cannot be considered scientifically valid.

All opinions in this report are stated to a reasonable degree of scientific certainty, and the methods used are among those that I employ in my professional work as a mineralogist.

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IARC (2012) defines the minerals that can be asbestos as follows: "the generic commercial designation for a group of naturally occurring mineral silicate fibres of the serpentine and amphibole series. These include the serpentine mineral chrysotile [], and the five amphibole minerals – actinolite, amosite [], anthophyllite, crocidolite [], and tremolite." International Agency for Research on Cancer (2012), *Monographs on the Evaluation of Carcinogenic Risks to Humans Vol. 100C: Asbestos (Chrysotile, Amosite, Crocidolite, Tremolite, Actinolite and Anthophyllite)*, 219.

II. BACKGROUND AND QUALIFICATION

I studied geology at Wellesley College, where my thesis involved field work to create a geologic map of the nearby Broadmoor Wildlife Sanctuary. I received my Ph.D. from the Massachusetts Institute of Technology in geochemistry and mineralogy. My Ph.D. thesis, entitled "Crystal chemistry and statistical analysis of iron in mineral standards, micas, and glasses," focused on minerals from a cross-section of geological problems. I was next a member of the Research Faculty at the California Institute of Technology, before holding faculty positions in the Department of Geological Sciences at the University of Oregon, Department of Geology and Astronomy at West Chester University, and most recently, at Mount Holyoke College, where I began in the Department of Geology and Geography before moving to chair the Department of Astronomy. I now hold an endowed chair as Kennedy-Schelkunoff Professor of Astronomy. I am also a member of the graduate faculty at the University of Massachusetts in Amherst, and a Senior Scientist at the Planetary Science Institute.

Over the course of my years in academia, I have taught mineralogy at least 20 times, including as a teaching assistant at MIT and at all three academic institutions where I have been a faculty member. I have supported public outreach related to mineralogy through the creation of two software programs used in many US states for teaching mineralogy to K-12 students. They include *The Study of Minerals* (1997) and *Hands-On Mineral Identification* (1999). I have also authored two textbooks: *Mineralogy and Optical Mineralogy: A Three-Dimensional Approach* with Mickey E. Gunter (first published in 2008, now in its 2nd edition and also available as an iBook) and *Geostatistics Explained, An Introductory Guide for Earth Scientists* with Steven McKillip (2010).

My research has resulted in more than 250 papers published in peer-reviewed journals, and it spans a number of different topics in mineralogy. Areas of interest have included metamorphic rocks from western Maine, tourmaline mineralogy from worldwide occurrences, mineralogy of rocks (xenoliths) from 90 km below Earth's surface (brought up by volcanoes), and many other rock types. The study of amphibole minerals has been a constant theme throughout my career, starting with a chapter in my thesis, extending to studies of its chemical variation that continue to this day. I have also published several papers relating to the mineralogy of asbestiform minerals, including papers on serpentine and amphibole asbestiform minerals.

See Dyar (1984) American Mineralogist, **69**, 1127-1144; Dyar et al. (1996), in Dyar et al., EDXA, Mineral Spectroscopy: A Tribute to Roger G. Burns, Special Publication #5, Geochemical Society, 273-289. Delaney et al. (1996), in Dyar et al., EDXA, Mineral Spectroscopy: A Tribute to Roger G. Burns, Special Publication #5, Geochemical Society, 170-177; King et al. (2000) Earth and Planetary Science Letters, **178**, 97-112. Gunter et al. (2003) American Mineralogist, **88**, 1944-1952; Minitti et al. (2008) Earth and Planetary Science Letters, **266**, 46-60; Minitti et al. (2008) Earth and Planetary Science Letters, **266**, 288-302; Sanchez et al. (2008) European Journal of Mineralogy, **20**, 1043-1053; Lupulescu et al. (2009) Canadian Mineralogist, **47**, 909-916; McCanta et al. (2009) Geochimica et Cosmochimica Acta, **72**, 5757-5780; Gunter et al. (2011) American Mineralogist, **96**, 1414-1417; Dyar et al. (2016) American Mineralogist, **101**, 1171-1189; Chan et al. (2016) Canadian Mineralogist, **54**, 337-351; Mueller et al. (2017) European Journal of Mineralogy, **29**, 167-180; Di Guiseppe et al. (submitted) Toxicology and Applied Pharmacology.

O'Hanley and Dyar (1993) *American Mineralogist*, **78**, 391-404; O'Hanley and Dyar (1998) *Canadian Mineralogist*, **36**, 727-739; Gunter et al. (2003) *American Mineralogist*, **88**, 1944-1952; Gunter et al. (2011) *American*

The unifying theme of my research is developing and using analytical techniques to solve geological problems, most of which involve identifying and quantifying minerals in mixtures. NASA is especially interested in this problem because many techniques that are used on planetary surfaces and in orbits involve acquiring signatures of rocks and then extracting individual mineralogy from those mixed signals. For example, in my work as a Participating Scientist on the science team for the Mars Exploration Rover *Curiosity*, we studied the problem of identifying individual minerals based on rock measurements.⁶ I studied meteorites from Mars and examined the spectra signatures of individual minerals and also the rock from which they were separated.⁷ We sometimes even create "fake rocks" from mineral mixtures and then make measurements of the mixtures to try to understand how to determine abundances of those minerals.⁸ My research depends heavily on the use of robust statistical analyses, and I often utilize machine learning techniques to solve problems in an unambiguous way.⁹

I work hard to uphold the standards of my profession by reviewing research proposals and journal publications continuously. I have been an Associate Editor for the *American Mineralogist* since 2000, handling up to 30 papers each year. I have edited one book for the Geochemical Society and one special volume of the *American Mineralogist*. I personally review at least a dozen manuscripts for a wide variety of journals every year, as well as many research proposals. I serve on review panels for the National Science Foundation and NASA almost every year, in which capacity I sometimes review as many as 18 proposals per year. As such, I am an experienced reviewer of scientific reports.

My research has been recognized with both national and international awards. I am a Fellow of the Mineralogical Society of America, the Geochemical Society and the Geological Society of America (GSA). I received the 2017 G. K. Gilbert award from the GSA for my outstanding contributions to planetary science and the 2018 Eugene Shoemaker Distinguished Scientist medal from NASA for "a scientist who has significantly contributed to the field of lunar and/or asteroid science throughout the course of their scientific career." I received the Hawley Medal from the Mineralogical Association of Canada in 2017, and currently hold the position of Helmholtz International Fellow with the German Aerospace Center (Deutsches Zentrum für Luftund Raumfahrt).

A copy of my current curriculum vitae is attached hereto as Exhibit A. My hourly rate for this work has been \$500 per hour.

Mineralogist, **96**, 1414-1417; Evans et al. (2012) American Mineralogist, **97**, 184-196; Swayze et al. (2018) American Mineralogist, **103**, 517-549; Di Guiseppe et al. (submitted) Toxicology and Applied Pharmacology.

Dyar et al. (2013) *Geological Society of American Annual Meeting*, Denver, Abstract #6-5; Dyar et al. (2014). *Lunar Planet. Sci.* **XLV**, Lunar Planet. Inst., Houston, CD-ROM #1788 (abstr.).

⁷ Dyar (2002) *LPI Conf.*, Houston, #6011; Dyar (2003) *Meteoritics and Planetary Science*, **38**, 1733-1752.

⁸ Carey et al. (2016) *Lunar Planet. Sci.* **XLVII**, Lunar Planet. Inst., Houston, (abstr.) #2626.

Boucher et al. (2015) *Journal of Chemometrics*, doi: 10.1002/cem.2727; Dyar et al. (2016) *Spectrochimica Acta B.*, **126**, 53-64; Boucher et al. (2015) *Spectrochimica Acta B*, **107**, 1-10; Carey et al. (2015) *Journal of Raman Spectroscopy*, **46**, 894-903; Boucher et al. (2017) *Journal of Chemometrics*, **31**, e2877; and Giguere et al. (2017) *Applied. Spectroscopy*, **71**, 1457-1470.

III. BACKGROUND: WHAT IS AN ASBESTIFORM MINERAL?

The definition of asbestos involves three different characteristics of a particle: **chemical composition**, **crystal structure and physical properties**.

By way of overview, the asbestiform nature of asbestos minerals is seen in **Figure 1**, which shows photomicrographs of tremolite and anthophyllite – two of the amphibole minerals that will form the basis for much discussion in this report.

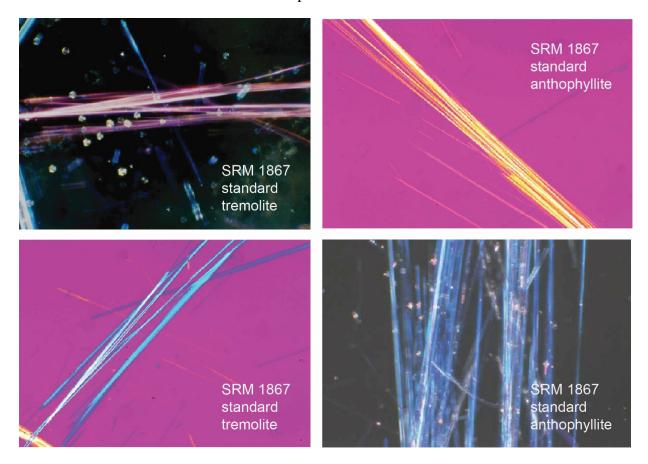


Figure 1. Microscopic images of tremolite (left) and anthophyllite (right) asbestos reference standards from the U.S. National Institute of Standards and Technology. All of these particles meet the standard definition of "asbestiform" articulated in Gunter (2010) and can "be separated lengthwise into fibers." Images from ISO 22262-1, Figures D.23, D.25, D.32, and D.34.

Each of these minerals has a specific chemical composition, crystal structure and physical properties. This is what makes them asbestos.

Thus, this Introduction explains the criteria relevant to the study of potentially asbestiform minerals in talcum powder. There are three issues to consider:

- **1. Mineralogy**: Are the mineral species among those that include asbestos?
- **2. Physical properties (habit)**: Do the minerals have an 'asbestiform' habit in other words, is this the asbestiform version of a mineral and not the non-asbestiform version?
- **3. Concentration**: What are the abundances of the minerals?

A. Mineral Species Identification

Summary: Many minerals have very similar chemical formulas, and many minerals occur with fibrous shapes, but "asbestos" minerals have *both* a specific chemical composition and a unique 'habit' or morphology.

The strict definition of a mineral species is that it (1) occurs in nature, (2) is stable at room temperature, (3) is generally produced by inorganic processes, (4) can be represented by a **chemical formula**, and (5) has a **specific, ordered arrangement of atoms**. The last two of these are especially important to this report.

Mineralogists use very specific mineral species names, regulated by an international committee, to assure that minerals with the same name have all five of these characteristics in common. There are currently ~5,500 known mineral species, and many more are found every year. Mineral classification is hierarchical, like biological classifications. Instead of the familiar "kingdom, phylum...genus, species" used in biology, mineralogists use mineral class, type, group and species. Species names are unique and highly specific. While there are **only six regulated asbestiform mineral species**, shown in red in **Table 1**, there are many other minerals related to them.

[Table 1 on next page.]

Mineral species names and compositions are certified by the Commission on New Minerals and Mineral Names (CNMMN) of the International Mineralogical Association.

Table 1. Minerals Similar to Talc that Contain Si, Mg, Fe, Ca, Na, H and O*

Mineral Species	Mineral Class	Chemical Formula	(Mg,Fe): Si
Anthophyllite	Amphibole	$Mg_7Si_8O_{22}(OH)_2$	7:8
Antigorite	Serpentine	$Mg_3Si_2O_5(OH)_4$	3:2
Chesterite	inosilicate >1 chain	Mg ₁₇ Si ₂₀ O ₅₄ (OH) ₆	17:20
Chrysotile	Serpentine	Mg ₃ Si ₂ O ₅ (OH) ₄	3:2
Clinohumite	Nesosilicate	$(Mg,Fe)_9(SiO_4)_4(OH)_2$	9:16
(clino)jimthompsonite	inosilicate >1 chain	$Mg_5Si_6O_{16}(OH)_2$	5:6
Enstatite	Pyroxene	Mg ₂ Si ₂ O ₆	1:1
Grunerite	Amphibole	Fe ₇ Si ₈ O ₂₂ (OH) ₂	7:8
Hydroxylclinohumite	Nesosilicate	Mg9(SiO4)4(OH)2	9:16
Cummingtonite	Amphibole	Mg7Si8O22(OH)2	7:8
Minnesotaite	sheet silicate	(Fe,Mg) ₃ Si ₄ O ₁₀ (OH) ₂	3:4
Sepiolite	modulated layers	Mg ₄ Si ₆ (OH) ₂₆ H ₂ O	4:6
Talc	sheet silicate	Mg ₃ Si ₄ O ₁₀ (OH) ₂	3:4
Tremolite	Amphibole	Ca ₂ Mg ₅ Si ₈ O ₂₂ (OH) ₂	5:8
Actinolite	Amphibole	$Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$	5:8
Ferrowinchite	Amphibole	CaNaFe ₄ (Al,Fe)Si ₈ O ₂₂ (OH) ₂	5:8
Richterite	Amphibole	Na(CaNa)Mg ₅ Si ₈ O ₂₂ (OH) ₂	5:8
Ferrorichterite	Amphibole	Na(CaNa)Fe ₅ Si ₈ O ₂₂ (OH) ₂	5:8
Riebeckite	Amphibole	$Na_2Fe^{2+}_3Fe^{3+}_2Si_8O_{22}(OH)_2$	5:8
Magnesioriebeckite	Amphibole	$Na_2Mg_3Fe^{3+}_5Si_8O_{22}(OH)_2$	5:8
Aegirine-augite	Pyroxene	(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al) ₂ O ₆	1:1
Diopside	Pyroxene	CaMgSi ₂ O ₆	1:1
Hedenbergite	Pyroxene	CaFeSi ₂ O ₆	1:1

Table 1. Formulas from the approved IMA "List of minerals" (http://nrmima.nrm.se/). Species names in red are termed "asbestos" when they occur in an asbestiform morphology. Shaded species in gray contain Na or Ca.

As shown above, these minerals all contain Si and cations such as Mg, Fe and Ca. Each has a particular ratio of Si to the other cations (the far-right column), which is important in the context of an energy-dispersive X-ray (EDXA) analysis (discussed below).

The mineral species significant to this report are those that combine the elements silicon (Si), magnesium (Mg), iron (Fe), calcium (Ca) and sodium (Na) along with hydrogen (H) and oxygen (O). Notice that the minerals are grouped in two groups, with Ca- and Na-bearing samples in the bottom half of the table. Many minerals have closely related (in some cases, identical) formulas; yet only those highlighted in red are mineral species that are regulated. Thus, it becomes critically important to discriminate these minerals carefully by chemistry and by crystal structure. Tools for these analyses fall into these two groups: tests of mineral composition (the chemical formula criterion noted above) and tests of determination of crystal structure (atomic arrangement). In addition, a particle must have a fibrous morphology to be considered asbestos.

B. Asbestiform Habit

Summary: Asbestos is defined as one of six particular minerals exhibiting the characteristics of an asbestiform habit, meaning they can be separated into flexible fibers with high tensile strength. Drs. Longo and Rigler confuse "counting criteria" with this definition, using a disingenuous distinction between what they call the "geological definition" and what they wrongly invent as the "regulatory definition."

Asbestos is a **noun** describing a specific material with a distinctive shape that is **asbestiform** (**adjective**), which means the crystal can be separated into flexible fibers with high tensile strength.

IARC (2012) defines the minerals that can be asbestos as follows: "the generic commercial designation for a group of naturally occurring mineral silicate fibres of the serpentine and amphibole series. These include the serpentine mineral chrysotile [], and the five amphibole minerals – actinolite, amosite [], anthophyllite, crocidolite [], and tremolite." IARC (2012), p. 222, also states: "Asbestos fibres tend to possess good strength properties (e.g. high tensile strength, weak and friction characteristics); flexibility (e.g. the ability to be woven); excellent thermal properties (e.g. heat stability, thermal, electrical and acoustic insulation); absorption capacity; and, resistance to chemical, thermal and biological degradation." Thus, there is agreement in the scientific community that asbestos exhibits fibrosity, high tensile strength and flexibility. This is repeated over and again in the published literature, for example:

Zoltai, T., "Amphibole Asbestos Mineralogy," Reviews in Mineralogy & Geochemistry, (1981) 9(5): 237-278 (stating "the individual asbestos fibers and acicular cleavage fragments are strikingly different. The fibers are extremely strong and flexible, while the acicular cleavage fragments are weak and brittle.").

Zoltai, T., "Amphibole Asbestos Mineralogy," Reviews in Mineralogy & Geochemistry (1981) 9(5): 237-278, at 272 (concluding that "cleavage fragments of amphibole crystals should yield the opposite strength-diameter effect than that observed in asbestos fibers").

McCrone, W.C., *The Asbestos Particle Atlas 36* (1980) (describing chrysotile as having "distinctive fine flexible fibrils").

Gary, M., McAfee, R., and Wolf, C., Eds., (1974) *Glossary of Geology* ("Asbestos is a commercial term applied to a group of silicate minerals that readily separates into long, thin, strong fibers that have sufficient flexibility to be woven, are heat resistant and chemically inert, are electrical insulators, and are therefore suitable for uses ... where incombustible, nonconducting, or chemically resistant material is required.").

Veblen D.R. and Wylie A. (1993) Reviews in Mineralogy, 28, 61-138 ("the asbestiform habit has a number of characteristics that differentiate it from other habits, but the main differences are the fibrillar structure and the fiber flexibility and strength").

Dyar and Gunter (2008) *Mineralogy and Optical Mineralogy* ("there are several types of minerals than can have asbestiform habits, which are distinguished by their fibrillar structure and the flexibility and strength of the fibers."

Asbestos Fibers and Other Elongate Mineral Particles: State of the Science and Roadmap for Research, (2011) U.S. Dept. of Health & Human Services, NIOSH, Current Intelligence Bulletin 62 at 6 (stating that "[t]he fibers of all varieties of asbestos are long, thin and usually flexible when separated"), 9 (stating that "[i]t has been suggested that crystals grown in an 'asbestiform' habit can be distinguished by certain characteristics, such as parallel or radiating growth of very thin and elongate crystals that are to some degree flexible, the presence of bundles of fibrils, and, for amphiboles, a particular combination of twinning, stacking faults, and defects"), 34 (describing chrysotile fibers as "consist[ing] of aggregates of long, thin, flexible fibrils that resemble scrolls or cylinders"), 36 (stating that "[a]sbestiform amphibole fibers consist of aggregates of long, thin, flexible fibrils that separate along grain boundaries between the fibrils").

Asbestiform Fibers: Nonoccupational Health Risks, National Research Council (1984) at 33 (stating that asbestos particles that have sufficiently small diameters have great strength and flexibility, and that the tensile strength of commercial quality asbestos fibers is 20 to 50 times greater than that of the non-asbestiform crystals of the same minerals), 38 (noting that the flexibility of asbestos fibers enables them to bend without breaking, which facilitates their passage through the respiratory tract into the deep lung), 41 (explaining that, "Asbestiform fibers, including asbestos fibers, are mineral fibers that are characterized by a specific set of interdependent physical properties, including fiber-like shape, enhanced strength and flexibility, increased durability, strong and defect-free surface structure, and the dependence of these properties on conditions of growth. The fiber properties that have been considered for possible association with deleterious health effects are respirability (i.e., fibers <3 µm diameter), size and aspect ratio, durability, flexibility and tensile strength, chemical composition, surface area, and surface charge.").

Gunter, M., *Defining Asbestos: Differences between the Built and Natural Environments*, 60 CHIMIA No. 10, 747, 748 (2010) (stating that a mineralogist would require that a fiber would need to exhibit a certain set of physical properties, i.e., be curved and flexible, in order to be classified as asbestos).

This usage is echoed in other documents, such as the methodology standards upon which Drs. Longo and Rigler purport to rely:¹¹

ISO 22262-1, -2, -3: defining asbestiform as a "specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility." ISO 22262-1 (2012) at 2; ISO 22262-2 (2014) at 8; ISO 22262-3 (2016) at 2.

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See 40 C.F.R. Ch. I (7-1-03 Edition) (defining asbestiform as "a specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility"); 40 C.F.R. 763.83 (defining asbestos as the "asbestiform varieties of" six minerals).

ISO 13794: defining asbestiform as a "specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility." ISO 13794 (1999) at 2.

ISO 10312: defining asbestiform as a "specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility." ISO 10312 (1995) at 2.

AHERA: defining asbestiform as a "specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility." 40 CFR Ch. 1, Pt. 763 Subpt. E, App. A, 728 (July 1, 2013).

ASTM D5755: defining asbestiform as "a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral." ASTM D5755 – 09 at 518-19.

ASTM D5756: defining asbestiform as "a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral." ASTM D5756 -02 (2008) at 532-33.

Further, if a particle is deemed to be fibrous, it does not mean it is asbestiform, as noted, for example, in Campbell et al. (1997), which recognizes that "the term fibrous has been used ... to describe all kinds of minerals that crystallize in habits resembling organic fibers... asbestiform is a specific type of fibrosity."¹²

Drs. Longo and Rigler appear to agree that asbestos has these qualities, although Dr. Longo calls this a "mineralogical definition." In Longo then distinguishes this from what he calls a "regulatory definition." That is misleading, because in reality, Drs. Longo and Rigler are referring to the **counting criteria** in regulatory documents, not what asbestos is or is not. Indeed, some of the very documents that they use to draw this disingenuous distinction expressly **define** asbestos according to what Drs. Longo and Rigler want to call the "mineralogical definition," *e.g.*, ISO 22262. Along the same lines, there are other criteria used under air sampling methodology (EPA

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¹² Campbell et al. (1997) *Information Circular 8751*. U.S. Bureau of Mines.

^{01/07/19} Longo Depo. 79:4-11 ("[asbestos] has to have high tensile strength, flexible splayed fibers, for asbestiform, and that's a geological definition."); 10/31/18 Longo Depo. 43:16-20 ("Q. And some of the features of asbestos growing in the asbestiform habit, as a geologist would use the term, have to do with tensile strength and flexibility; correct? A. That is the geological definition"); 10/24/18 Longo Depo. 269:13-23 ("Q. So I believe you said that the definition of asbestiform habit that you just gave is not your definition, is that what you said? A. That's correct. I mean, it's the standard definition, it's fibrous. Now, what it also says for asbestiform, that it has to have high tensile strength, it has to have splayed ends, it has to have this, it has to have that. That's a geological definition . . ."); 2/05/2019 Longo Depo. 222:2-17 ("Q. Do you agree with ISO 13794 that this method cannot discriminate between individual fibers of the asbestos and nonasbestos analogs of the same amphibole material? A. ... but, no, it does not meet the geological definition for asbestos, high tensile strength, flexible, and so on and so forth.").

Asbestos Hazard Emergency Response Act, AHERA¹⁴ and EPA 600/R-93¹⁵) to describe fibers. In those documents, mean aspect ratios for populations of asbestos particles range from 20:1 to 100:1, which is inconsistent with the findings from Drs. Longo and Rigler. Certain protocols may look to whether the aspect ratios of detected particles are at least 3:1 for measurements by polarizing light microscopes¹⁶ and at least 5:1 for TEM measurements.^{17,18}

In other words, these aspect ratio dimensions alone do not imply that the observed particles are asbestos – there are non-asbestiform amphiboles and many other minerals that have aspect ratios above 3:1 or 5:1. Drs. Longo and Rigler confirmed this in their depositions. ¹⁹ This is discussed in more depth below in Section VII.

C. Measurements of Concentration

Summary: An appropriate analytical technique must be used to determine the degree to which there is asbestos in a talc sample, depending on the level of any impurity.

If asbestos minerals are present in talcum powder, it is important to be able to assess their abundance. There are spectroscopic methods, such as visible, Raman, and infrared spectroscopies, that work well on such powders, but they cannot detect minor phases that are <~1%. Particles that are present in very low abundances (such as parts per million) are nearly impossible to quantify. The measurement of small amounts of asbestos as an impurity in talc in greater detail in Section V, below.

US Code Title 15, Chapter 53, Subchapter II. Asbestos Hazard Emergency Response, https://www.govinfo.gov/content/pkg/USCODE-2011-title15/pdf/USCODE-2011-title15-chap53-subchapII.pdf.

EPA/600/R-93/116, https://tinyurl.com/y4z765xp.

⁴⁰ C.F.R. 763, Subpart E, https://www.govinfo.gov/content/pkg/CFR-2009-title40-vol30/pdf/CFR-2009-title40-vol30-part763.pdf.

EPA/600/R-93/116, https://tinyurl.com/y4z765xp.

⁴⁰ C.F.R. 763, Subpart E, https://www.govinfo.gov/content/pkg/CFR-2009-title40-vol30/pdf/CFR-2009-title40-vol30-part763.pdf.

^{2/5/19} Longo Depo. 246:5-15 ("Q. Okay. Is there anything in the published literature that you've seen that suggests that there are cleavage fragments with a greater than 5-to-1 aspect ratio? A. There's been a number of published articles that state things like that, yes. Q. Are there any published articles that state that there are cleavage fragments that have greater than 3-to-1 aspect ratio? A. Yes, there is publications that state that."); 2/6/19 Rigler Depo. 191:21-193:4 ("Q. Using a geological definition of asbestos, can you have a cleavage fragment that is greater than 5-to-1 aspect ratio? A. In my opinion, the answer to that is yes. Q. Okay. And using the geological definition of asbestos as you have used it, there can be an asbestiform particle that has an aspect ratio of below 3-to-1? A. Are you talking about what kind of particle? Q. Asbestiform particle. A. Smaller than 3-to-1? Q. Yeah. ... in most cases of that size, you know, you may see some that are in that range, but you have to use the PLM to see them, probably.").

IV. MINERAL IDENTIFICATION USING CHEMICAL COMPOSITION: EDS

Drs. Longo and Rigler opine on the results of analyses done by their five analysts using electron dispersive spectroscopy (EDS or EDXA for x-ray spectroscopy). ²⁰ This section discusses the use of EDS to the identification of mineral compositions.

A. How EDS Works

Summary: EDS, when properly used, can provide a limited analysis of the *chemical makeup* of certain mineralogical samples.

In this technique, an electron source is used to produce high-energy electrons that are focused onto a tiny spot on a sample. The electrons interact with the atoms in the crystal structure, causing a low-energy inner shell electron to be ejected from the atom. Higher-energy electrons from higher shells in that atom quickly drop down to fill the void, and in doing so, they release energy in the form of x-rays. The energy is characteristic of each element and reflects the spacing of the two shells involved, which is unique to each element. A detector counts how many electrons are scattered and at what energies. This produces a graph of energy (x axis) vs. counts (y axis) as shown in **Figure 2**.

[Figure 2 on next page.]

Both Drs. Longo and Rigler testified that they did not personally run any of the experiments on which they

February 1, 2019 Report, at 4 ("MAS [i.e., Longo and Rigler] sent a number of historical J&J samples to J³ Resources for both PLM and XRD analysis using ISO 22262-1 and ISO 22262-3 protocols.").

2/5/19 Longo Depo. 258:16-18 ("Q: [T]here were four people doing analysis in the MDL report; right? A: Correct.");

opine in their reports. Instead, they were involved in various levels of oversight and supervision. *See* 2/6/2019 Rigler Depo. 13:6-15 ("Q. Did you ever use a PLM for the purposes of this report? A. No, I did not. Q. Did you ever use a TEM for the purposes of this report? A. Not for the purposes of this report. Q. Did you ever use an XRD device for the purposes of this report? A. We do not have the XRD device or that type of device at our laboratory."); 11/27/18 Longo Depo. 447:6-10 ("Q. Okay. For any of the analyses that were done for the November 14, 2018, report, were you the analyst that reported the findings on the bench sheets? A. No."). There were five such analysts: four at Drs. Longo and Rigler's laboratory, and one at J3, a laboratory not formally affiliated with Drs. Longo and Rigler. *See*

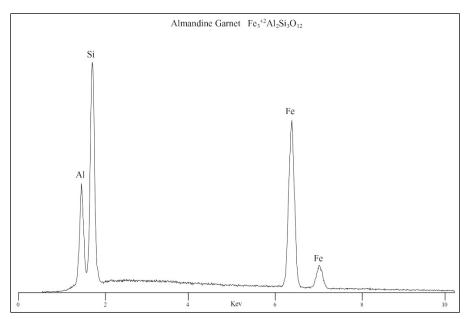


Figure 2. EDS spectrum of the mineral species almandine ($Fe_3Al_2Si_3O_{12}$) from the classic book by Severin (1984).²¹

Peaks in the EDS plots arise from individual elements, and the areas of the peaks are roughly proportional to the amounts present (e.g., Severin, 1984). In the example above, the mineral is almandine, which has a nominal formula of Fe₃Al₂Si₃O₁₂. There are three formula units of Si and three formula units of Fe. Notice how the total area of the Fe peaks is roughly comparable to the total are of the single Si peak because both elements are present in roughly equal proportions. There are only two formula units of Al, and that peak is thus somewhat smaller.

This example was purposely chosen to be simple. But in most EDS spectra, the correspondence between peak areas and elemental abundances is not so direct due to a host of factors²² (see Section B below). There are ways to correct for them, and every TEM instrument sold today includes a software package based on the same physical principles that can make the appropriate corrections via detailed calculations. Such calibrations are used to convert the areas to true (albeit semi-quantitative) elemental percentages in the mineral. Independent software is also available for this purpose (e.g., Desktop Spectrum Analyzer II²³ or XIDENT²⁴), which allows a user to import spectral files with a standard format and extract quantitative information from them.

Depending on the x-ray intensity and the type of detector used, some elements are difficult to measure by EDS. EDS **cannot** be used to quantify either hydrogen (H) or oxygen (O) accurately.

Severin (1984) Energy Dispersive Spectrometry of Common Rock-Forming Minerals, Kluwer.

Deriving quantitative information from an EDS spectrum is a complicated process due to instrumental (such as the type of window on the detector), experimental (which energy was used), and physical (absorption and fluorescence) factors. A nice summary of this is given at https://cstl.nist.gov/div837/837.02/epq/dtsa2/.

https://cstl.nist.gov/div837/837.02/epq/dtsa2/.

Rhoades (1976) *XIDENT – A computer technique for the direct indexing of electron diffraction spot patterns* (Research Report 70/76), https://ir.canterbury.ac.nz/handle/10092/11515.

Therefore, distinguishing among the minerals in talcum powder similar to talc (**Table 1**) requires that magnesium (Mg), iron (Fe), calcium (Ca) and silicon (Si) be distinctive for each phase and have x-ray peaks in the right energy region (0-10 keV), as seen in **Figure 2** above. EDS analyses are generally considered to be semi-quantitative for chemical analyses, with errors of about $\pm 5\%$ relative for major elements (Si, Al, Ti, Mg, Fe, Ca, Na, and K). Attaining such accuracy requires use of a calibration and ideal experimental conditions.

B. Factors Affecting Accuracy of EDS

Summary: EDS can distinguish many minerals from one another, but it has significant limitations when minerals from completely different groups have similar chemistries. For example, EDS cannot accurately distinguish anthophyllite *amphibole* from talc *sheet silicate* (or even enstatite *pyroxene*), or tremolite *amphibole* from diopside *pyroxene*.

Mineral identification based on EDS data alone is quite dependent on coaxing the best possible chemical data out of a semi-quantitative instrument. To a first order, the areas of each peak in an EDS spectrum reflect the concentration of that element.²⁷ But as noted above, other factors, such as particle geometry, spectrometer model, type of detector, ²⁸ detector sensitivity, and the efficiency of x-ray generation, come into play as well to affect the peak areas, requiring sophisticated calibration to obtain interpretable results. **Figure 3** shows the EDS pattern of cummingtonite from the classic book of EDS patterns by Severin (1984). This mineral has a composition of (Mg,Fe)₇Si₈O₂₂(OH)₂.²⁹ Even though this sample has more Mg than Fe atoms, the peak areas are radically different due to the factors noted above: 5% for Mg and 26% for Fe. **In other words, the peak areas are not directly proportional to the abundances of the elements.**

[Figure 3 on next page.]

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Newbury and Rotchie (2015) *Journal of Material Sciences*, **50**, 493-518; Miller and Mirtič (2013) *Geologija*, **56**, 5-17.

Hafner, Energy Dispersive Spectroscopy on the SEM: A Primer, http://www.charfac.umn.edu/instruments/eds_on_sem_primer.pdf.

Severin (2004) Energy Dispersive Spectrometry of Common Rock Forming Minerals. Springer, Kluwer.

As noted by Dr. Longo in deposition, EDS instruments can have Si-drifted, Li-drifted, or windowless detectors. 2/5/19 Longo Depo. 101:8-23. He continues: "if it's a light element detector, the magnesium can be a little higher, the silicon will be your primary peak, somewhere in the 25-30 percent of the magnesium for a non-light element detector. And the calcium peaks and the magnesium peaks are usually very similar in size." 2/5/19 Longo Depo. 77:17-78:6.

The ideal "end member" composition of cummingtonite is $Mg_7Si_8O_{22}(OH)_2$, but it forms a continuous series with grunerite (Fe₇Si₈O₂₂(OH)₂) so it can contain up to 3.5 formula units of Fe. In the formula given above, the "Mg,Fe" is a notation that indicates that there is more Mg than Fe.

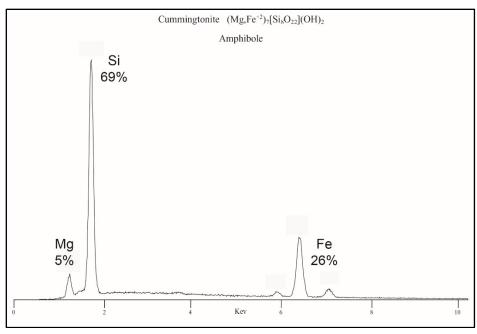


Figure 3. EDS spectrum of the mineral species cummingtonite, $(Mg,Fe)_7Si_8O_{22}(OH)_2$, from Severin (1984). ³⁰

When proper calibrations are used, EDS has ±5% or larger error bars, making it only semi-quantitative under the best of protocols (i.e., not sensitive enough for subtle chemical differences). This can be a significant limitation on the usefulness of EDS in many contexts. For example, one way scientists use EDS readouts to roughly determine the chemical makeup of a sample is to use the ratio of the peak areas of the elements (e.g., Mg+Fe) to Si. The right-hand column of **Table 1** shows these ratios in some rock-forming minerals containing Si, Mg, Fe and Ca. Those data suggest that only accurate analyses will allow these minerals to be distinguished on the basis of chemistry alone, given the error bars of EDS. The most accurate methods (such as electron probe microanalysis, EPMA, which has a beam size ~1000-20,000 nm) can measure mineral chemistry well enough to tell these minerals apart, but these methods lack the excellent spatial resolution of EDS on a TEM, which is roughly 0.5 nm (~1000× smaller than EPMA). Therefore, studies of asbestos-bearing rocks generally use EDS for chemical analyses, sacrificing accuracy for the ability to focus the beam on a tiny particle.

By way of specific example, **Figure 4** shows the Mg/Si ratios for selected **Table 1** minerals, along with propagated error bars of 5%, which are generally accepted best accuracies for analyses

As noted on the previous page, the ideal "end member" composition of cummingtonite is Mg₇Si₈O₂₂(OH)₂, but it forms a continuous series with grunerite (Fe₇Si₈O₂₂(OH)₂) so it can contain up to 3.5 formula units of Fe. In the formula given above, the "Mg,Fe" is a notation that indicates that there is more Mg than Fe. Figure from Severin (1984) *Energy Dispersive Spectrometry of Common Rock-Forming Minerals*, Kluwer.

Newbury and Ritchie (2015) *Journal of Material Sciences*, 50, 493-518; Miller and Mirtič (2013) *Geologija*, 56, 5-17.

Severin (1984) 23 (it is possible to "determine the approximate relative concentrations of the element. The number of characteristic X-rays is, to a first order, related to the number of atoms of a particular element in the sample...").

of particles and rough surfaces.³¹ The **dashed** purple line in **Figure 4** shows that anthophyllite and enstatite are identical within the error bars. The **dotted** purple line shows that talc and sepiolite are indistinguishable. Within these error bars, talc and anthophyllite are also identical because their error bars overlap (thick purple shaded line).

The analogous conclusions can be drawn from the diagram at right for tremolite (amphibole) and diopside (pyroxene), where the y axis is now (Mg+Ca)/Si. Again, these minerals have completely different crystal structures, yet their formulas are quite similar. The purple shaded bar here indicates how much these two minerals overlap. These two minerals would be indistinguishable to EDS, even though they belong to completely different mineral groups (amphibole and pyroxene, respectively) with very distinct properties.

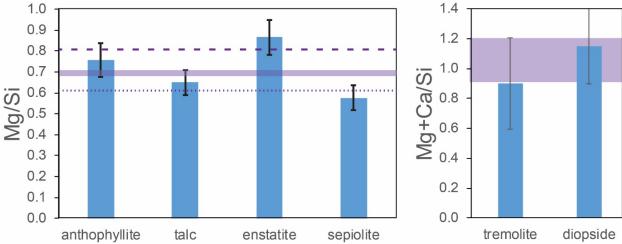


Figure 4. Ratios of the weight percentage of magnesium (Mg) to silicon (Si) along with propagated 5% errors on each measurement.³³ Formulas from webmineral.com with 5% errors added.

Further, the EDS results in the Longo/Rigler MDL Reports labelled as "tremolite" may very well be consistent with minerals other than diopside. Drs. Longo and Rigler have never produced their quantitative data and, accordingly, this analysis cannot be completed.³⁴

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For example, anthophyllite contains 28.78 wt.% Si and 21.79 wt.% Mg. EDS determines these values to within $\pm 5\%$, i.e., 28.78 ± 1.44 and 21.79 ± 1.09 . So Mg/Si = 0.65 and the propagated error on Mg/Si is ± 0.08 . The same calculation is done here for talc, enstatite, sepiolite, tremolite, and diopside.

For example, these may include at least monticellite, bredigite, merwinite and rondorfite, which are other minerals that contain only Si, Mg and Ca. Many more common mineral species would be included in this list if Fe and Na were allowed as possible chemical constituents.

C. The Analysts Used By Drs. Longo And Rigler Do Not Use EDS In A Way Accepted By The Scientific Community, Resulting In Unsupportable Conclusions

Summary: The "eyeball" method of estimating EDS data employed by Drs. Longo and Rigler is not considered reliable by the scientific community. Drs. Longo and Rigler do not report the quantitative data one needs to make scientifically valid conclusions, and their conclusions are therefore unsupportable.

It is clear that Dr. Longo does not understand the limitations of EDS results that are explained above. He states, "I believe every spectra in here is quantitative EDS analysis" (2/5/19 Longo Depo. 72:22-23). He explains how his analysts use peak ratios (like those above) to identify minerals based on EDS spectra. To identify tremolite from anthophyllite, "[y]ou have a magnesium and calcium peak that are pretty close. Typically the calcium peak can be a little lower. ... the magnesium can be a little higher, the silicon will be your primary peak, somewhere in the 25 to 30 percent of the magnesium ... [a]nd the calcium peaks and the magnesium peaks are usually very similar in size. And then we look at the amount of iron to see if we're going to call it actinolite or tremolite. And not aware of any other minerals out there that have those ratios, so that's how I call it tremolite." (2/5/19 Longo Depo. 77:14-78:6).

It is apparent from these remarks that Dr. Longo, who admits he has never taken any courses in mineralogy (2/5/19 Longo Depo. 16:2-3), is unaware of the breadth of other possible minerals that can match these EDS patterns within the known analytical errors. His methodologies are contrary to those that are accepted in the scientific community.

D. EDS Data from Drs. Longo And Rigler Show Great Variabilty

Turning now to EDS data in the Longo/Rigler MDL Reports, **Figure 5** shows several different talc spectra. Note the variable amounts of Fe in the sample, and the varying ratio between the intensities of the Mg and Si peaks. Do these grains really have different compositions, or are the variations from analytical imprecision? It would only be possible to make this determination with calibrated data.

[Figure 5 on next page.]

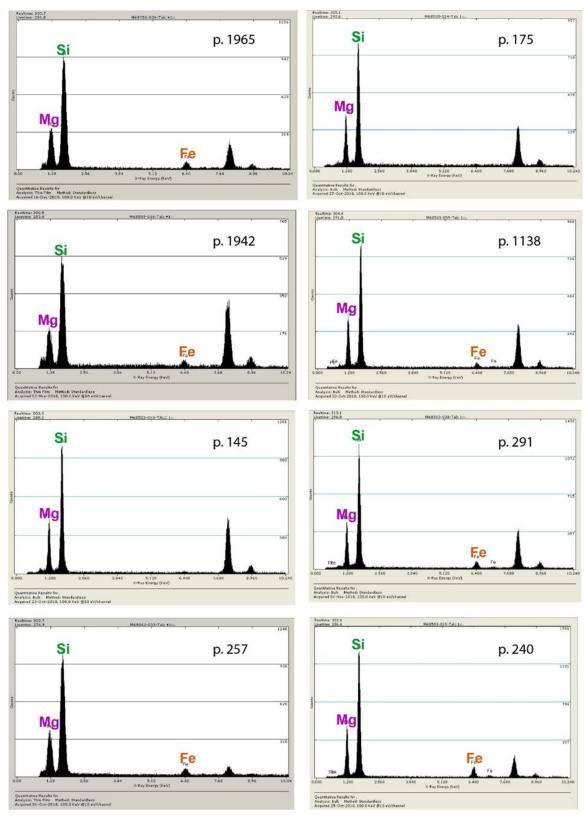


Figure 5. Talc spectra in the January 2019 Longo/Rigler MDL report, showing variable ratios between the intensity/area of the Mg and Si peaks. Note also the varying amounts of Fe in these talcs.

Similarly, **Figure 6** shows a number of different anthophyllite spectra from the January 2019 MDL Report. Although these are all identified as "anthophyllite," they could be almost any mineral containing Mg and Fe but no Ca (cf. top portion of **Table 1**).

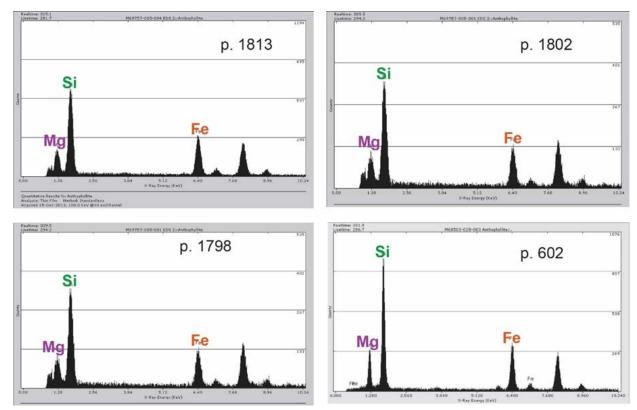


Figure 6. Anthophyllite spectra in the January 2019 Longo/Rigler MDL report showing Fe peaks that are larger than Mg peaks.

In fact, these spectra in **Figure 6** closely resemble the spectrum of cummingtonite shown in **Figure 3**. Without a calibration to correct the peak areas for experimental variables, it is quite difficult to determine which minerals are present. Moreover, the formula of anthophyllite, (Mg,Fe)₇Si₈O₂₂(OH)₂) should have much more Mg than Fe, which is clearly not the case here.

It is **nearly impossible to tell some of these spectra apart** solely by visual inspection of their EDS patterns. Calibrations are needed to produce optimal, albeit semi-quantitative, results. These graphs show why the calibration procedure to convert from peak areas to elemental concentrations is routinely part of the software that comes with a TEM instrument. ³⁵ It is irresponsible for Drs. Longo and Rigler to choose not to employ calibration software to assist with their mineral identifications.

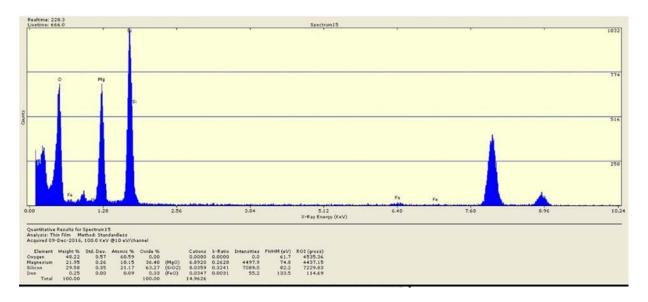
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This calibration is done using first principles calculations or by comparison with spectra of a small number of standards with known compositions, as described in Newbury (1995) "Standardless" quantitative electronic probe microanalysis with energy-dispersive x-ray spectrometry: Is it worth it? *Anal. Chem.*, **67**, 1866-1871.

E. Drs. Longo And Rigler And Their Analysts Appear to Have Deliberately Avoided Reporting The Data That Would Actually Be Useful Here

Summary: EDS instruments and their accompanying standard software are capable of produce numerical data on chemical composition. Drs. Longo and Rigler appear to have deliberately avoided producing or reporting those data.

The more information extracted from an EDS analysis, the more a scientist can narrow down the composition of the subject mineral. A typical EDS output is shown in **Figure 7**, with each peak identified on the screen and the quantitative analysis given below the spectrum. Results include a listing of each element in weight %, the standard deviation of that number based on repeat analyses, ³⁶ atomic %, and cations (positively charged ions), along with the total weight % of the summed elements.



Quantitative Analysis: Th Acquired 09	in Film M	ethod: Stan	dardless	annel						
Element	Weight %	Std. Dev.	Atomic %	Oxide %		Cations	k-Ratio	Intensities	FWHM («V)	ROI (gress)
Oxygen	40.22	0.57	60.59	0.00		0.0000	0.0000	0.0	61.7	4535.36
Magnesium	21.95	0.26	10.15	36.40	(MgO)	6.8920	0.2628	4497.9	74.0	4437.15
Silicon	29.58	0.35	21.17	63.27	(SiO2)	8.0359	0.3241	7089.0	82.2	7229.83
Iron	0.25	0.00	0.09	0.33	(FeO)	0.0347	0.0031	55.2	133.5	114.65
Total	100.00			100.00		14.9626				

Figure 7. Mostly amphibole EDS spectrum from the Connecticut Geological Society using the eZAF Smart Quant calibration, from their web site at https://www.geologicalsocietyct.org/geoconnections-articles/recent-analyses-of-connecticut-amphiboles.

Asked whether their EDS tools could produce this information, Dr. Rigler agreed. Looking at the precise readout that is in **Figure 7** above, he testified:

Notice that accuracy is not given in the output – only precision ("Std. Dev." in the table below the figure).

Q. Okay. It is from your report. So is that what an EDXA spectra looks like?

A. Yes.

Q. And you'll notice on the bottom left-hand corner it says elements and it has got some elements and it says total?

A. Yes.

Q. Your software can generate information that fills in that; correct?

A. Yes. ³⁷

The quantitative data are conspicuously lacking from all the EDS patterns in the Longo/Rigler MDL Reports. It is also curious that the printouts of EDS patterns from the Longo/Rigler MDL Reports list the elements chosen by the analyst (Mg, Si, etc.), followed by the word "Total," which implies that a column of numbers to the right of the element list gives a listing of peak areas. In the more typical EDS analysis from another lab, as seen in **Figure 7**, the composition is given in cations as $Si_{8.04}Mg_{6.98}Fe_{0.03}$. It would be very difficult to obtain these numbers simply by looking at the graph.

Yet the EDS protocol in the lab of Drs. Longo and Rigler does exactly that – the analysts "eyeball" the spectra and decide which mineral is present based on the peak areas.³⁸ This practice cannot be justified because it is highly subjective. Compositions are not given for any of the EDS patterns produced by Drs. Longo and Rigler, even though their EDS is equipped to provide this data.

Dr. Longo states that the quantitative numbers are "not something that's required to render my opinion in this case."³⁹ He also states that "you can't look at the areas, but the peak ratios is what's important here."⁴⁰ This practice of identifying mineralogy by visual inspection of EDS data may be defensible in other applications undertaken at Drs. Longo and Rigler's lab **where the material being tested is already known**. In such cases, EDS is used simply to distinguish among the known minerals. But "eyeballing" of compositions is completely inappropriate in talc testing because of the many different minerals that could be present and how similar they may appear.

Mineral identifications (like those in **Figure 7**) might have been easier if quantitative peak area data had been presented in the Longo/Rigler MDL Reports. It is a standard capability of EDS software to generate elemental composition data (literally one click of a button), but those data are missing from the Longo/Rigler MDL Reports. Had the list of peak areas for individual elements been included in the reports, these data could have corroborated the mineral species identifications made by the other techniques (e.g., PLM by Lee Poye at J3 Resources, Inc.). But because those

³⁷ 2/6/19 Rigler Depo. 55:23-56:7 (emphasis added).

³⁸ 2/5/19 Longo Depo. 81:18-83:14; 2/6/19 Rigler Depo. 96:24-97:12.

³⁹ 2/5/19 Longo Depo. 83:15-20.

⁴⁰ 2/5/19 Longo Depo. 76:10-14.

data are missing, little useful information can be gleaned from the EDS plots beyond the presence/absence of Ca.

F. Lack Of Data On And Misuse Of Standards

Summary: Dr. Longo testified that his analysts used "reference standards" of asbestiform minerals in their analysis, but Drs. Longo and Rigler did not produce any, and no such standards exist that could match the Longo/Rigler "eyeball" methodology.

In his deposition, Dr. Longo stated that "they routinely check reference samples," ⁴¹ although none of the reports say this. ⁴² This lack of data on reference standards makes it impossible to assess the accuracy of the SAED analyses in the Longo/Rigler MDL Reports. Dr. Longo further states that his lab has asbestos reference samples "on the TEM walls." ⁴³ In other words, in the laboratory of Drs. Longo and Rigler, analysts match the asbestos EDS patterns on their screens to ones printed out and hanging on the walls. Because the only reference standards used by the analysts are for asbestos minerals, the confirmation bias is apparent. It virtually guarantees that a particle they identify on the basis of EDS patterns will be asbestos, regardless of whether it really is.

G. Species Identifications Vary By Date And By Analyst

Summary: The mineral species identifications in the Longo/Rigler MDL Reports are not only scientifically indefensible, they appear demonstrably biased according to the particular EDS operator.

Because the mineral species identifications in the Longo/Rigler MDL Reports were made by naked-eye review of the EDS spectra, it would likely be difficult for different analysts to produce comparable results, and in fact, it was. **Figure 8** shows the distribution of mineral species by analyst. Despite the fact that most of these individuals worked on the project over time from 2018-2019 and the samples were randomly selected, ⁴⁴ the **results appear to be biased by each operator's perceptions of peak intensities**. For example, Lee Poye identifies only anthophyllite. Mehrdad Motamedi and Anthony Keaton consistently find more tremolite than anthophyllite, and Jayme Callan finds more anthophyllite. It is difficult to understand how this could happen if the samples they were testing were randomly distributed.

[Figure 8 on next page.]

⁴¹ 2/5/19 Longo Depo. 41:10-18.

⁴² 2/5/19 Longo Depo. 45:9-12.

⁴³ 2/5/19 Longo Depo. 41:10-18.

⁴⁴ 2/5/19 Longo Depo. 45:13-20 ("Q: How does your lab distribute samples to individual analysts to test? Is it random? Is it like some analysts get a certain kind of sample? A: It's random. Q: Is that the same for J3? Did you give them random samples? A: Random samples.").

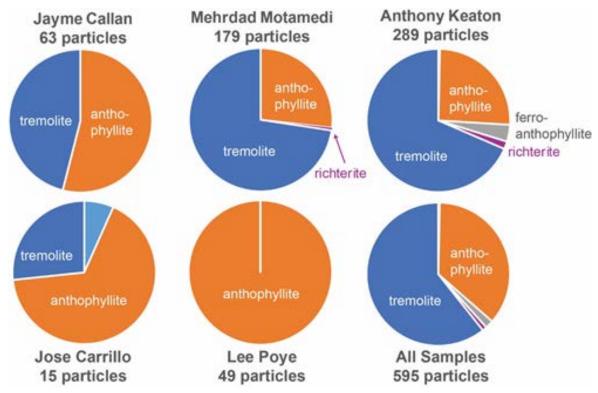


Figure 8. Distribution of mineral species identifications sorted by analyst for all samples reported to date. ⁴⁵

If all samples were assigned to analysts at random, then the distribution of mineral species should all be the same.

Perhaps the distribution of mineralogy seen in **Figure 8** was the result of which localities each analyst happened to receive? For example, data in the Longo/Rigler MAS Reports indicate that samples mined from Vermont appear to have ~75% anthophyllite and 25% tremolite. So both Lee Poye and Jose Carrillo, who analyzed only Vermont samples (**Figure 9**), should have identical 75/25 ratios of anthophyllite/tremolite, and they do not.

[Figure 9 on next page.]

This compilation includes all grains for which mineral species were identified with dimensions (i.e., TEM data) in each of four of the reports: 1) Supplemental Expert Report & Analysis of Johnson and Johnson Baby Powder and Valeant Shower to Shower Talc Products for Amphibole Asbestos March 11, 2018, 2) Analysis of Historical Johnson's Baby Powder M69042 October 2018, 3) The Analysis of Johnson & Johnson's Historical Baby Powder & Shower to Shower Products from the 1960's to the Early 1990's for Amphibole Asbestos November 14, 2019, and 4) The Analysis of Johnson & Johnson's Historical Product Containers and Imerys's Historical Railroad Car Samples from the 1960's to the Early 2000's for Amphibole Asbestos January 15, 2019. This analysis thus includes data on 163 samples, with some repetition.

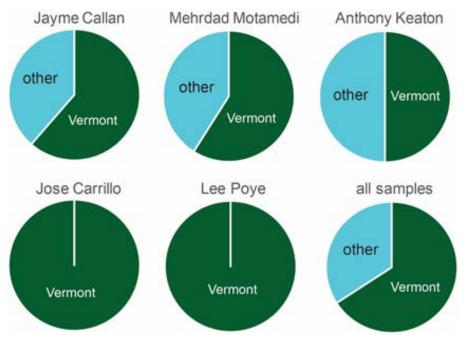


Figure 9. Distribution of mineral species identifications from Vermont vs. all other localities, sorted by analyst for all samples reported to date.

In other words, there is no justifiable explanation for why some analysts find different numbers of each mineral species. The logical conclusion is that the mineral species identifications are inconsistent and non-repeatable.

Furthermore, **Figure 10** shows that species identifications also appear to change over time.

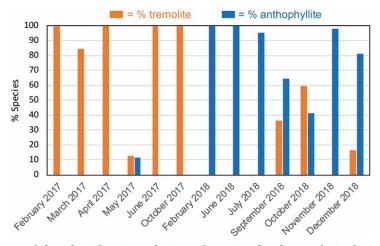


Figure 10. Comparison of the identification of mineral species by the analytical team of Drs. Longo and Rigler over time. Colors and data sources as in Figure 8.

Most of the mineral species identified in 2017 were tremolite, while most of the sample species identified in 2018 were anthophyllite. This could only happen if the samples analyzed in 2017 were all from the same tremolite-bearing locality. But this would be unlikely if all samples were randomly tested.

These variations cast doubt over *all* EDS-based species assignments made by this team, and therefore make it unlikely that any asbestos mineral species could be identified on the basis of these EDS data. This conclusion is of no surprise given the analytical uncertainties of the technique, as explained above.

H. Conclusions Regarding EDS

- The generally accepted error bars on EDS chemical analysis indicate that it is impossible to easily discriminate amphibole (or any other minerals with similar compositions) species from talc using this technique. The compositions of a wide range of Si-Mg-Si minerals are so similar that EDS, especially qualitative EDS as used here, cannot distinguish among them.
- EDS spectral printouts alone are insufficient to discriminate any particular mineral species. It is impossible to tell anything quantitative about these compositions without calibrated results, which could easily have been produced by Drs. Longo and Rigler or their assistants using software already built into their instruments. Instead, the analysts "eyeballed" the mineral identifications, and compared their spectra only to known asbestos minerals without considering other possible phases that could have similar EDS data.
- Drs. Longo and Rigler and their analysts thus used a flawed methodology that resulted in wildly inconsistent results across different analysts and over time. In science, proof involves demonstrating that a result is replicable. Because the Longo/Rigler MDL Reports show no actual data (i.e., percentages of each element derived from a calibration) of any kind to substantiate their mineral species assignments, these results are not credible and cannot be scientifically considered.
- EDS patterns alone cannot determine the morphology of a mineral particle. Even if a EDS pattern can be matched with a specific mineral, this only identifies which mineral species is present. Such analysis cannot determine whether or not the mineral is asbestiform.

V. MINERAL IDENTIFICATION USING CRYSTAL STRUCTURE: SAED

The alternative to analyses from EDS is to identify minerals based on their crystal structure - i.e., the arrangement of the atoms. Analytical approaches to this problem generally exploit how energy in the form of light or x-rays passes through the arrangement of atoms in a crystal. The success of any technique in identifying a specific mineral species is dependent on the distinctiveness of each species' crystal structure.

A. Introduction To Selected Area Electron Diffraction (SAED)

Summary: SAED, when properly used, can be used to create patterns of dots. When properly analyzed, these patterns can identify the *crystal structure* of the sample. This is done using the "d-spacing," corresponding to spaces between the dots.

A transmission electron microscope (TEM), the same instrument used for EDS, is employed to acquire SAED patterns. For SAED, the waves of electrons pass through the crystal structure and interact with the atoms, which act as a diffraction grating. Some electrons travel straight through the sample and others are scattered by the atoms. By using each mineral's unique crystal structure, the amount by which the electrons are diffracted can be calculated. In the resultant pattern of dots (e.g., **Figure 11**), **spacings between dots are inversely proportional to the spacings between atoms**. ⁴⁶ Thus, the arrangement of dots can be diagnostic of a mineral structure.

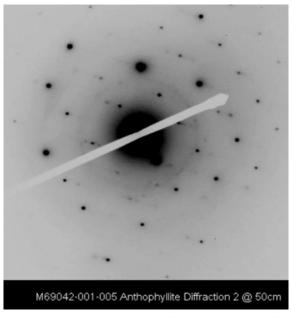


Figure 11. SAED pattern of M9042-001-005 from page 64 of the 2019.1.16 Longo/Rigler MDL report.

Optimal SAED patterns are acquired when the individual crystal happens to be lying with a zone axis parallel to the electron beam. This is most easily done when a crystal exhibits elongate (like an amphibole) or sheet-like (sheet silicates) morphology. To obtain these special

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⁴⁶ Dyar and Gunter (2008) Mineralogy and Optical Mineralogy. Mineralogical Society of America.

orientations, most instruments allow the stage to be tilted to produce an optimal pattern, but this requires considerable skill and time.⁴⁷ In other words, the crystal must be oriented to obtain a zone axis pattern in which the reflections line up to produce an interpretable pattern.

B. To Uniquely Identify An Unknown Mineral, SAED Patterns Should Be Taken From Multiple Axes

Summary: Multiple SAED patterns along multiple "zone axes" are required to uniquely identify a mineral.

For an accurate identification of an unknown mineral, at least two (and preferably more) SAED patterns must be taken with the sample oriented along a known zone axis.⁴⁸ Consider this simplified analogy: imagine a cube, with an atom at every corner, and in the middle of each face (**Figure 12**).⁴⁹

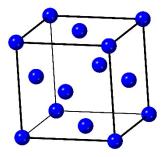


Figure 12. Crystal structure of copper, which is simply a cube with atoms at the corners and in the middle of each face. Viewed at this angle, the pattern of atoms can be easily discerned.

If the cube is held with only one side facing the viewer, the corners would line up, and it would look something like **Figure 13**, with its SAED pattern shown at right.

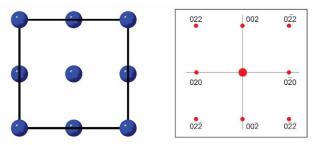


Figure 13. Crystal structure of copper, with a face of the cube facing the viewer.

The resultant SAED pattern of the orientation in **Figure 13** is shown to the right. From this perspective, it is impossible to tell where all the atoms are – do they lie on the edges and corners,

⁴⁷ ISO 22262-1 document, page 65.

⁴⁸ Yamate, EPA (1984).

This example happens to be the structure of pure copper, taken from Wyckoff R W G (1963) Second edition. Interscience Publishers, New York, New York, cubic closest packed structure. *Crystal Structures* 1:7 - 83.

or on faces and corners, or both? How tall is this shape? Is it a cube or a rectangle with a square end? Viewed from only one angle, these questions cannot be answered.

So a view from a different angle, as seen in **Figure 14**, is needed to confirm where all the atoms are located. Now it can be seen that this is a cube with atoms at the corners and in the center of each face.

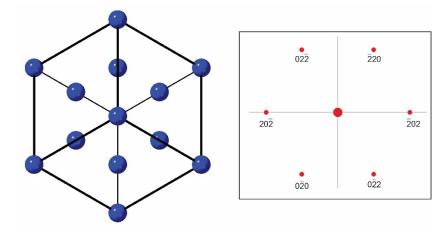


Figure 14. Crystal structure of copper, viewed along a diagonal with opposite corners held vertical to the viewer. The resultant SAED pattern is shown at right.

This example highlights the fact that a combination of views from different angles is needed to determine the full structure. Most minerals are far more complicated, and SAED patterns are correspondingly complex, but this is the gist of why multiple SAED images are required to identify a mineral.

By way of example, the patterns in **Figure 15** are the three SAED patterns for tremolite looking down the three zone axes. Note that each image is different. Each image taken in isolation can correspond to other minerals, ⁵⁰ but together these three uniquely identify tremolite.

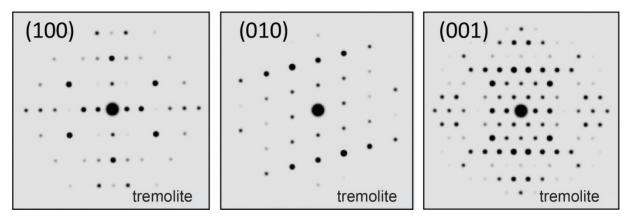


Figure 15. Simulated SAED patterns for tremolite projected onto different crystallographic orientations, indicated by the numbers in parentheses in each pattern. Adapted from Figure 15.2 in Dyar and Gunter (2008).

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⁵⁰ Yamate, EPA, 1984.

The difference between a real SAED pattern (**Figure 11**) and a simulated one (**Figure 15**) demonstrates the importance of operator skill in acquiring these patterns.

It is not a simple process to align a sample on a zone axis parallel to the electron beam. The ISO 22262-1 document, page 65, explains it like this:

"Rotate the sample until the fibre image indicates that the fibre is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fibre is at the eucentric position. Tilt the fibre until an ED pattern appears which is a symmetrical, two dimensional array of spots. The recognition of zone-axis alignment conditions requires some experience on the part of the operator. During tilting of the fibre to obtain zone-axis conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections, the possibility of twinning or multiple diffraction exists, and some caution should be exercised in the selection of diffraction spots for measurement and interpretation. It is important to recognize that not all zone-axis patterns that can be obtained are definitive."

C. SAED Images In The Longo/Rigler MDL Reports Are Of Very Low Quality And Are Poorly Documented

Summary: The Longo/Rigler MDL Reports use visual inspection of low-quality and poorly documented images, which cannot uniquely identify minerals unless one has already concluded one is looking at asbestos *prior* to the SAED analysis.

As can be seen in examples from one of the Longo/Rigler MDL Reports (2018.1.16) in **Figure 16**, not all of the SAED patterns in the Longo/Rigler MDL Reports look like anything close to those in **Figure 15**. Most of these patterns are inconclusive and inconsistent, depending on the orientation of each crystal and its likely mineralogy.

[Figure 16 on next page.]

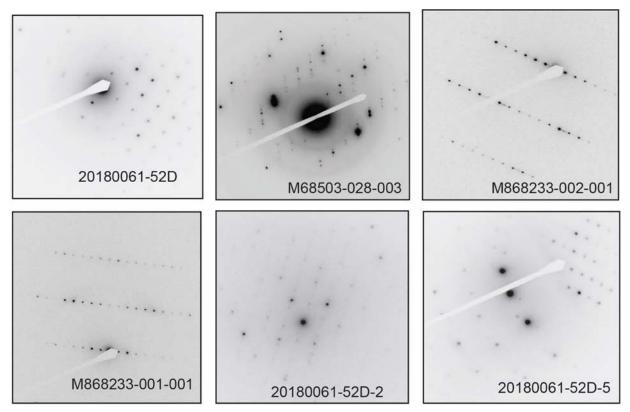


Figure 16. SAED patterns for "anthophyllite" from the 2019.1.16 Longo/Rigler MDL Report.

Most of these images are of such low quality that they can only be used to indicate that this is a crystalline material. With only a few exceptions, ⁵¹ the reports do not indicate at what orientation each image was taken. Finally, spacings and angles between the dots are given only for a handful of samples given in the *Diffraction Verification* documents (see below). The SAED patterns are labeled with mineral species names using only visual inspection based on operator experience, ⁵² a methodology for which the Longo/Rigler MDL Reports cite no support. This practice may be able to distinguish among species for materials that are already known to contain asbestos, but it may fail in the applications where the spectrum of possible mineralogy is broad. The use of only visual inspection shows a lack of understanding of the complexity of identification of these minerals using electron diffraction.

In the *Diffraction Verification* documents, some zone axes are indicated, but only for a subset of particles examined. In the main reports, no information on orientation is given.

⁵² See 2/5/19 Longo Depo. 246:22-47:4.

D. The SAED Patterns In The Longo/Rigler MDL Reports Do Not Uniquely Identify Tremolite Or Anthophyllite

Summary: The *d*-spacing information given for the SAED patterns in the Longo/Rigler MDL Reports does not uniquely identify tremolite and anthophyllite because their methodology is flawed and inadequate.

It is not clear that Drs. Longo and Rigler's analysts even used the *d*-spacing values for their SAED images. Drs. Longo and Rigler testified that the *d*-spacing numbers that they produced (in their *Diffraction Verification* documents) were only used **after** the mineral identification as a way of "verifying" results.⁵³ If true, Drs. Longo and Rigler did not interpret the SAED patterns in a manner consistent with generally acceptable scientific principles, and indeed, Drs. Longo and Rigler do not cite any publication for interpreting a SAED report without using the *d*-spacing. Dr. Rigler testified that these numbers were generated after the initial expert reports were served,⁵⁴ but then changed his testimony when confronted with the implications of this (that conclusions were made without consulting *d*-spacings).⁵⁵

Either way, the *d*-spacing values in the Longo/Rigler MDL Reports do not allow identification of tremolite and anthophyllite. Specifically, in the *Diffraction Verification* documents provided by Drs. Longo and Rigler, SAED patterns are "read" from 219 patterns from six samples by measuring and calculating the spacings between rows of dots. In those patterns, the space between the lines was measured and then converted to units of angstrom (\mathring{A})⁵⁶ using an **unspecified constant.** Lacking knowledge of that constant, *d*-spacings cannot be easily verified for the patterns in the Longo/Rigler MDL Reports. ⁵⁷

Take the following **Figure 17** as a specific example of a Longo/Rigler SAED pattern, to exemplify one of the many problems with their SAED analysis:

[Figure 17 on next page.]

⁵³ 2/6/19 Rigler Depo. 116:22-118:17; 2/5/19 Longo Depo. 182:16-183:11.

⁵⁴ 2/6/19 Rigler Depo. 118:6-14.

⁵⁵ 2/6/19 Rigler Depo. 127:2-129:3.

The diffraction camera constant is unique to each particular TEM instrument, as well as the magnification and focal length used to obtain the pattern, and is expressed in units of mm \times Å. It is divided by the distance between two adjacent dots to arrive at the *d*-spacing values. According to Dr. Rigler, the lab uses calculations based on lattice parameters of gold to calibrate their SAED patterns. 2/6/19 Rigler Depo. 114:20-115:3.

In the *Diffraction Verification* documents, the camera constant is given, but it is specified in units of pixels. Because the images have been scanned, it is impossible to apply that constant to the images, and thus the spacings cannot be verified.

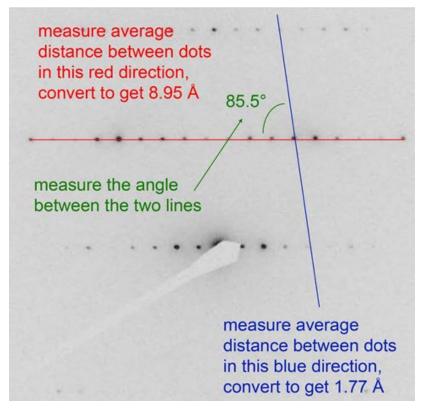


Figure 17. SAED pattern of M68233-001-001 from p. 693 of the 2019.1.16 Longo/Rigler MDL Report (colored annotation added).

For the example shown in **Figure 17**, the relevant values are d(hk0) = 8.95 Å, d(hk1) = 1.77 Å, and $\theta = 85.5^{\circ}$. Next, it is necessary to find those three values in "look-up tables." Below is the only look-up table that Drs. Longo and Rigler used (found on each "Diffraction Verification" page):

Amphibole Type	Pg. #	Card #	Calculated Spancing	Range +/- 5%
Grunerite	449	31-631	5.2	4.94 - 5.46
Actinolite	4	25-157	5.13	4.87 - 5.39
Tremolite	1192	13-437	5.09	4.84 - 5.34
Crocidolite	993	19-1061	5.19	4.93 - 5.45
Anthophyllite	48	9-455	5.28	5.02 - 5.54

This means that **Drs. Longo and Rigler used tables only for the five recognized asbestos amphibole species and therefore did not check to see if their values corresponded to non-asbestiform minerals.** ⁵⁸ To the extent Drs. Longo and Rigler already concluded they were looking at asbestos prior to the SAED analysis, as strongly implied by the table, their analysis was predetermined and conclusion-oriented, not scientific.

⁵⁸ 2/6/19 Rigler Depo. 101:2-17.

E. Drs. Longo And Rigler Use Incomplete *d*-Spacing Data

Summary: Use of only one d-spacing value, as reported by Drs. Longo and Rigler and their analysts, is almost never diagnostic of any mineral. Indeed, their d-spacing calculation can correspond to $\sim 25\%$ of all common rock-forming minerals, not one specific mineral.

As explained with the analogy using a cube, above, **finding only one** *d***-spacing is only rarely diagnostic of any mineral** because minerals are three-dimensional. In the *Diffraction Verification* documents from Drs. Longo and Rigler, each page has a listing at the top that shows **one** of the calculated *d*-spacings for each of the five amphiboles:

Amphibole Type	Pg.#	Card #	Calculated Spancing	Range +/- 5%
Grunerite	449	31-631	5.2	4.94 - 5.46
Actinolite	4	25-157	5.13	4.87 - 5.39
Tremolite	1192	13-437	5.09	4.84 - 5.34
Crocidolite	993	19-1061	5.19	4.93 - 5.45
Anthophyllite	48	9-455	5.28	5.02 - 5.54

These pages then go on to provide a single "Calculated Spacing Å." For example, there are 31 pages of patterns for sample M69757, for which only **one** *d*-spacing has been calculated. No zone axis information is presented.

The problem here is that providing a single d-spacing is not diagnostic of any specific mineral species, or even mineral group. A search of the mineral database ⁵⁹ from Dyar and Gunter (2008) shows that 93 out of 369 (25%) mineral species have at least one d-spacing of 4.94 to 5.46 Å. A more comprehensive analysis using the American Mineralogist Crystal Structure database shows more than one thousand crystal structures that have at least one d-spacing in the range above. ⁶⁰ Finally, a search was made of the Inorganic Crystal Structure Database, ⁶¹ which contains 203,830 inorganic crystal structures (minerals and chemical compounds). In that database, 8%, 7%, and 6% of the structures had a, b, or c, respectively in the range of 4.94 to 5.46 Å. Some of these are shown below in Table 2, where it is apparent that many different minerals, many of which are not amphiboles, have a unit cell dimension of 5.3 Å in some direction. Therefore, deriving a 5.3 Å spacing from an SAED pattern would be completely inconclusive. A single unit cell dimension (d-spacing) is almost never diagnostic enough to uniquely identify a mineral species.

The Mineral Database search engine and database, which is sold on the Apple web site at https://itunes.apple.com/us/app/mineral-database/id815681529?mt=12, was designed to allow searches on a database consisting only of common rock-forming minerals. The list of minerals with at least one *d*-spacing of 4.94 to 5.46 Å includes (in addition to the minerals listed in **Table 2**) such common minerals as aragonite, calcite, hematite, ilmenite, biotite and phlogopite mica, pyrite and quartz.

The database is found at http://rruff.geo.arizona.edu/AMS/amcsd.php, for minerals under Cell Parameter and Symmetry. Searching each of the *a*, *b* and *c* axes for *d*-spacing between 4.94 and 5.46 Å produces 1897 records, 1227 records, and 2245 records, respectively. Note that these are individual crystal structure refinements, and multiple examples of different mineral species are included.

https://ucsd.libguides.com/crystallography/icsd

Table 2. Unit Cell Dimensions for Selected Relevant Mineral Species

Mineral	Formula	a (Å)	b (Å)	c (Å)
enstatite	MgSiO ₃	18.2	8.8	5.2
anthophyllite	Mg ₇ Si ₈ O ₂₂ (OH) ₂	18.5	18.0	5.3
magnesiocummingtonite	$Mg_7Si_8O_{22}(OH)_2$	9.5	18.2	5.3
sepiolite	$Mg_4Si_6O_{15}(OH)_2.6H_2O$	13.5	26.7	5.2
antigorite	$Mg_3Si_2O_5(OH)_4$	5.3	9.5	14.9
talc	$Mg_3Si_4O_{10}(OH)_2$	5.3	9.5	18.9
tremolite	$Ca_2Mg_5Si_8O_{22}(OH)_2$	9.8	18.1	5.3
actinolite	Ca ₂ (Mg,Fe) ₅ Si ₈ O ₂₂ (OH) ₂	9.8	18.1	5.3
ferrowinchite	$CaNaFe^{2+}_{4}(Al,Fe^{3+})Si_{8}O_{22}(OH)_{2}$	9.8	18.1	5.3
richterite	Na(CaNa)Mg ₅ Si ₈ O ₂₂ (OH) ₂	10.3	18.4	5.2
ferrorichterite	$Na(CaNa)Fe^{2+} Si_8O_{22}(OH)_2$	10.0	18.2	5.3
riebeckite	$Na_2Fe^{2+}_3Fe^{3+}_2Si_8O_{22}(OH)_2$	9.8	18.0	5.3
magnesioriebeckite	$Na_2Mg_3Fe^{3+}_2Si_8O_{22}(OH)_2$	9.8	18.0	5.2

In his deposition on February 6, 2019, Dr. Rigler acknowledged this problem. When asked, "Can you distinguish anthophyllite from cummingtonite with SAED alone?", Dr. Rigler acknowledged that "you'd have to do zone axis in a couple of different zones to tell." In his deposition, Dr. Longo confirmed this statement, saying that identification of anthophyllite requires measuring both "d-spacing and a second pattern from a different crystalline orientation." Despite this acknowledgment, Drs. Longo and Rigler only report two zone axes for a single anthophyllite grain, and give a single zone axis measurement for only two other samples (M69042-008 and M68503-001).

The testimony of Drs. Longo and Rigler confirms that their methodology does not identify specific minerals.⁶⁵

^{62 2/6/19} Rigler Depo. 108:23-109:18.

^{63 2/5/19} Longo Depo. 133:3-9.

These zone axes are provided in the *Diffraction Verification* documents. Several values of d(hk0), d(hkl), and θ are given for sample M69757-007, but no zone axis is derived. For sample M69042-002, several values are given for sample d(hk0) only. For sample M68233, four different zone axes are presented; this is the only sample for which more than one zone axis is given.

⁶⁵ 2/5/19 Longo Depo. 157:3-8 ("Q. Okay. What other amphibole in the Mineral Powder Diffraction File have *d*-spacing ranges that span 5.23? A. Most of your amphibole minerals, both monoclinic and orthorhombic, will have *d*-spacings in this range."); 2/6/19 Rigler Depo. 119:3-8 ("Q. Now, for this verification page, you calculated a *d*-spacing of 5.23; correct? A. Correct. Q. And that falls within the range of every single amphibole on that list; right? A. Correct."); *see also id.* 119:22-120:8.

F. Unfeasible d-Spacings In Longo/Rigler Diffraction Verification Documents

Summary: Some of the Longo/Rigler *d*-spacing interpretations have impossible values.

Lack of understanding of *d*-spacings is also shown by reports of indefensible *d*-spacings. Page 14 of the M69757 *Diffraction Verification* document demonstrates particularly poor methodology:

Can	nera K (pixelÅ)	Meas. Dist	ance (pixels)	Calculate Spacing (Å)
	810.7		411.6	1.97
Streaking Obse	erved:		Closely s	paced dots:
Type of amphil	oole diffraction v	erified:	Anthophyllite	
MAS Job #:	M69757-0	<u>07-001</u>		Film #: NA
Analyst: <u>JC</u>			Dat	te of Photo: 12/15/2018
Date Verified:	12/18/2018	3	E	OS Verified: <u>YES</u>
one Axis Infor	mation_			
i(hk0) =				
l(hki) =	1.97			
ingle =	83			
'A =				

Under the heading "Zone Axis Information," the d(hk0) value is indicated to be 10.1 Å. A user would then look for this value in the look-up tables. But as seen on the first page of the anthophyllite reference sheet shown below, there are no (hk0) values higher than 9.25 Å.

AN	ITH	OP	PHY	LL	ITE			d-S	Spa	cin	g (Å	A) and	d Interfaci	ial Angle	0 (°)		Page 1 of 32
						30									, Orthorhombic) (k+l)=2n for (0kl)]		
	Zor	ne A	Axis			(1	ı k	0)		(h	kΙ)	d(hk0)	d (hkl)	(hk0)/(hkl)	θ	To C-Axis(°)
]	0	1	0]	(2	0	0)	(1	0	1)	9.25	5.08	1.82	74.1	90.0
[0	1	1]	(2	0	0)	(0	1	1)	9.25	5.06	1.83	90.0	73.6
Ì	0	1	1	ĺ	(2	0	0)	(1	1	1)	9.25	4.88	1.89	74.7	7 73.6
Ī	0	1	0	ī	(2	0	0)	(2	0	1)	9.25	4.59	2.02	60.3	90.0
Ĩ	0	1	1	i	(2	0	0)	(2	1	1)	9.25	4.44	2.08	61.3	73.6
ĺ	0	1	2	i	(2	0	0)	(1	2	1)	9.25	4.42	2.09	76.2	59.5
Ī	0	1	2	i	(2	0	0)	(2	2	1)	9.25	4.08	2.27	63.8	59.5
j	0	1	0	ĺ	(2	0	0)	(3	0	1)	9.25	4.01	2.31	49.4	90.0
Ī	0	1	1	i	(2	0	0)	(3	1	1)	9.25	3.91	2.36	50.6	73.6
Ī	0	1	2	í	(2	0	0)	(3	2	1)	9.25	3.66	2.53	53.6	59.5
i	0	1	0	i	(2	0	0)	ì	4	0	1)	9.25	3.48	2.66	41.2	90.0
Ĭ	0	1	1	í	ì	2	0	0)	(4	1	1)	9.25	3.42	2.71	42.4	73.6
í	0	1	2	î	(2	0	0)	ì	4	2	1)	9.25	3.24	2.85	45.5	59.5
i	0	1	0	í	ì	2	0	o i	ì	1	0	21	9 25	2 61	3 54	81 9	

A second sample, M69042-008 particle 37, lists a *d*-spacing of 13.46. In these cases, **either** the measurement itself is bad or these cannot be anthophyllite, or both.

In addition, many of the other d-spacings calculated in the *Diffraction Verification* documents lie outside their own stated ranges for amphiboles: M69757-005 particle 23; M69757-007 particles 30 and 31, M6942-002 particles 12, 15, 16, 19, 20, 22; M68503-001 particle 7, M68503-002 particle 10, M68503-026 particles 35, 57, and 58. In some cases, the values are likely for (hkl), which also has a wide range of values at $1.7\pm5\%$ Å. But in most of these examples, the stated d-spacings are just anomalous and likely incorrect for any of the asbestiform amphiboles.

G. The *d*-Spacing For What Drs. Longo And Rigler Identify As Anthophyllite Actually Corresponds To One Of Two Other Minerals

Summary: The *d*-spacing for "anthophyllite" identified by the Longo/Rigler MDL Reports actually corresponds to cummingtonite or grunerite.

In a small number of instances, ⁶⁶ the Longo/Rigler MDL Reports identify the zone axis. For example, because the M68233-001-001 pattern in **Figure 17** was assumed to be anthophyllite (as indicated by the label on the image on page 693), the anthophyllite look-up table ⁶⁷ was employed. Part of the relevant page from that look-up table is given below.

Zone axes are identified in 41 particles from only 6 of the 70 samples in the Longo/Rigler MDL reports.

Look-up tables were created by Su (2003) *How to Use the d-Spacing/Interfacial Angle Tables to Index Zone- Axis Patterns of Amphibole Asbestos Minerals Obtained by Selected Area Electron Diffraction in Transmission Electron Microscope*, 2008 report, Asbestos Analysis Consulting.

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ANTHOPHYLL	(JCPDS 9-45	oacing (Å) an 5: a 18.5Å b 17.9Å ma permits only h=	c 5.28Å α 90	° \$\beta\$ 90° \$\beta\$ 90°,	, Orthorhombic)	F	Page 3 of 32
Zone Axis	(h k 0)	(h k l)	d(hk0)	d (hkl)	(hk0)/(hkl)	θ	To C-Axis(°)
[1 0 2]	(0 2 0)	(4 5 2)	8.95	1.93	4.64	57.4	60.3
[2 0 3]	(0 2 0)	(3 6 2)	8.95	1.88	4.75	50.9	66.8
[2 0 1]	(0 2 0)	(172)	8.95	1.83	4.90	44.4	81.9
[1 0 1]	(020)	(272)	8.95	1.80	4.97	45.2	74.1
[2 0 3]	(020)	(372)	8.95	1.76	5.08	46.5	66.8
3 0 1 1	(020)	(103)	8.95	1.75	5.11	90.0	84.6
1 0 0 1	(020)	(013)	8.95	1.75	5.11	84.4	90.0
i 3 0 1 i	(020)	(113)	8.95	1.74	5.13	84.4	84.6
3 0 2 1	(020)	(203)	8.95	1.73	5.18	90.0	79.2
1 3 0 2 1	(020)	(213)	8.95	1.72	5.20	84.5	79.2

There is no listing specifically for d(hk0) = 8.95, d(hkl) = 1.77, (hk0)/(hkl) = 5.07, and $\theta = 85.5^{\circ}$. Therefore, the analyst chooses something close, as highlighted in yellow. Importantly, the **85.5°** is not the same as 90°, as can be plainly seen in **Figure 17**. This indicates that the **SAED** pattern is **NOT** a match to anthophyllite, but likely from a different amphibole. Either the analyst mis-read the table or she/he used the angle relative to the c-axis instead of to θ . In either case, the methodology and the science are incorrect.

There are many amphibole minerals with similar compositions (**Table 1**). If a SAED pattern is not consistent with anthophyllite, then another amphibole file can be tried, such as that for the grunerite 68 mineral series, as given below. The yellow highlights in this table represent a good match to the M68233-001-001 SAED pattern. The d(hkl) and the angle are exact matches, suggesting that this mineral is from the cummingtonite-grunerite series. A different amphibole might give an even better match, but **there are no other look-up tables**.

This reliance on look-up tables that only exist for asbestiform mineral species virtually guarantees that an asbestos mineral will be identified. This is a serious flaw in methodology.

Minerals in the cummingtonite-grunerite series all have very nearly the same crystal structure (with slight variations due to the size difference between Mg^{2+} and Fe^{2+}), and thus share the same look-up table. These minerals have a very different structure from anthophyllite even though the formulas are similar. So the failure by Drs. Longo and Rigler's analysts to use the proper look-up table is an egregious error.

(BRU	JNI	ERI	TE		d -Spacing (Å) and Interfacial Angle $\boldsymbol{\theta}$ (°)								Page 1 of 23			
						(JC	PDS	19-16					30Å <i>c</i> 5.338Å opermits only (h -		6° y 90°, Monoclini tions]	c)	
	Zoi	ne .	Axis	S		(1	ı k	0)		(1	n k	1)	d (hk0)	d (hkl)	(hk0)/(hkl)	θ	To C-Axis(°)
į	2	0	-1	į	(0	2	0)	į	1	-5	2)	9.190	2.005	4.58	57.0	116.3
Ī	1	0	0	i	(0	2	0)	(0	6	2)	9.190	1.988	4.62	49.5	101.9
Ī	2	0	3	i	(0	2	0)	(3	5	-2)	9.190	1.914	4.80	58.6	57.2
Ī	1	0	-1	i	(0	2	0)	(2	4	2)	9.190	1.913	4.80	65.4	128.0
j	1	0	2	j	(0	2	0)	(4	-2	-2)	9.190	1.911	4.81	78.0	47.1
[3	0	1	1	(0	2	0)	(1	1	-3)	9.190	1.771	5.19	84.5	91.1
Ī	2	0	-1	1	(0	2	0)	(1	-7	2)	9.190	1.768	5.20	47.7	116.3
Ī	3	0	2	j	(0	2	0)	(2	0	-3)	9.190	1.754	5.24	90.0	80.3
Ī	3	0	2	i	(0	2	0)	(2	-2	-3)	9.190	1.723	5.33	79.2	80.3

This analysis shows that the analysists used by Drs. Longo and Rigler and their analysts incorrectly used the anthophyllite table to identify the zone axis in a particle that was likely from the cummingtonite/grunerite series. The same mismatch can be observed for all the values of d(hk0), d(hkl), and the interfacial angle θ compiled for anthophyllite in the report on sample M68233 entitled *Diffraction Verification*.

H. Drs. Longo And Rigler And Their Analysts Do Not Follow EPA Methodology, Including Confirmation On At Least Two Zone Axes

Summary: The EPA "Yamate III" methodology for confirming the presence of asbestos in talc requires *two* SAED zone axis determinations and an EDS analysis, which the Longo/Rigler MDL Reports do not provide.

The protocol for "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy" (Yamate, EPA, 1984) states in Section 2, "Conclusions and Recommendations" (p. 3) that there are three necessary levels of effort:

The EM methodology for measuring the concentration of airborne asbestos fibers has been refined and specified, and is recommended for field evaluation. The methodology is based on a TEM analytical protocol that is divided into three levels of effort: Level I, for screening many samples; Level II, for regulatory action; and Level III, for confirmatory analysis of controversial samples. The three-level analytical methodology is cost-effective, and will provide the required results for proper assessment of asbestos.

Level III is used in these cases (Section 6, Level III Asbestos Analysis, Discussion of Protocol, from p. 44):

The Level III protocol is an extension of the Level II analysis procedures described in Section 5. This extension may be necessitated by the need for positive identification of the specific amphibole species in situations where (1) fundamental disagreements between parties involved in a litigation require further clarification;

(2) for identification purposes, e.g., as causative agents in medical diagnosis or studies; (3) for quality control of Level II analysis in special situations, and/or; (4) for source samples, whether as bulk material or bulk-air type where a legal judgment is anticipated.

The protocol states that identification requires **two** SAED zone axis determinations and an EDS analysis. Dr. Rigler stated in his deposition that anthophyllite particles were double-verified by *d*-spacing analysis (2/6/19 Rigler Depo. 125:18-20). But out of **hundreds of particles** studied by Drs. Longo and Rigler (or their analysts), zone axis determinations are provided for only 41 particles in **six** samples. In fact, zone axis information is provided for only **one** "anthophyllite," M68233.⁶⁹ **As such, Drs. Longo and Rigler and their analysts do not follow EPA protocol**.

Indeed, both Dr. Rigler and Dr. Longo admit to problems inherent in using too few zone axis, despite doing it themselves.

I. Conclusions Relating to SAED Analysis

- SAED images in the Longo/Rigler MDL Reports are often of very low quality with poor documentation. Some of them are uninterpretable. The reports do not provide the calibration constant needed to convert dot spacings into atom spacings.
- Indexing of diffraction patterns using look-up tables is done incorrectly. The use of asbestos mineral species' look-up tables to determine zone axes presumes that the mineral species of the sample has already been identified. Even lacking such information, Drs. Longo and Rigler and their analysts consistently use the anthophyllite look-up table, and do not consider whether another mineral species might give a better match. "Anthophyllite" diffraction patterns indexed by Drs. Longo and Rigler do not match those in look-up tables for that mineral.
- Unfeasible *d*-spacings exist in the Longo/Rigler Diffraction Verification documents. In two of the 41 cases for which zone axis information is provided, the calculated *d*-spacing is impossible. In several other cases, the single *d*-spacing lies outside the stated range for amphibole minerals.
- Drs. Longo and Rigler and their analysts do not follow EPA methodology for "confirmatory analysis of controversial samples." Although Drs. Longo and Rigler both admitted in deposition that two zone axis determinations are necessary to confirm the identity of anthophyllite, zone axis data are provided for only 41 particles in six samples, and the requisite two zone axes on the same sample aliquot are given for only three samples.

Although Drs. Longo and Rigler (or their analysts) believe this particle to be anthophyllite, it is actually a mineral in the cummingtonite-grunerite series.

- Even when patterns are correctly acquired, the same mineral will have very different SAED patterns depending on its orientation.
- SAED patterns alone cannot determine the morphology of a mineral particle. Even if an SAED pattern can be matched with a specific mineral and orientation, this only identifies which mineral species is present. Such analysis cannot determine whether or not the mineral is asbestiform.

VI. MINERAL IDENTIFICATION USING PLM

The Longo/Rigler MDL Reports purport to use polarizing light microscopy ("PLM") to identify asbestos in talc. This section discusses the proper use of PLM, as compared to how it was used in plaintiffs' experts' reports.

A. How PLM Works

Summary: PLM, when properly used, can identify asbestos mineral content by observing the morphology and refractive indices of the particles in the sample.

PLM is a technique that uses a specially-equipped microscope to polarize the beams of light that pass through a sample (**Figure 18**). This "polarization" causes all the waves of light to vibrate in the same direction, creating a set of near-parallel light waves. When a sample is rotated on the microscope stage, the parallel waves of light interact with the sample differently depending on the angle between the layers of atoms in the sample and their spacings.

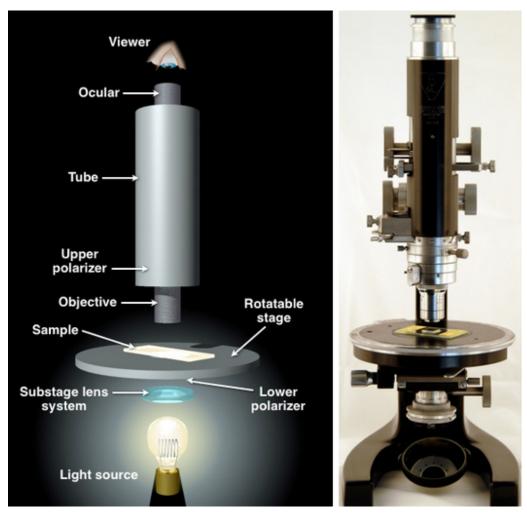


Figure 18. Photograph and sketch of a simple polarizing light microscope, from Dyar and Gunter (2008), Figure 17.2.

The polarizers above and below the microscope stage allow a user to study how parallel waves of light interact with rows of atoms in a crystal structure. Changes in appearance upon rotation can be diagnostic of specific mineral groups and, sometimes, mineral species.

The PLM method is, by Longo's own admission, "primarily used today for the analysis of asbestos-added products where the asbestos contents of these products are typically over 1% by weight." PLM allows mineral species to be identified using inherent properties of each mineral: the shape of the grains, the way they break and their densities.

Drs. Longo and Rigler and analysts examined 61 samples using PLM (ISO 22262-1 method), which requires that asbestos be identified using six types of observations:

- Morphology
- Color and pleochroism
- Birefringence
- Extinction charactertistics
- Sign of elongation
- Refractive index

The last property, refractive index (R.I.), is a ratio between the speed of light traveling through a vacuum and how fast it travels through (in this case) a mineral. The value of n is different in varying directions of a mineral, and so the change in refractive index with orientation can be diagnostic of a mineral group or species.

To measure the value of n in an unknown grain, it is surrounded by an oil with a known n, and the crystal is examined. For example, in the protocol used by Drs. Longo and Rigler, "three mounts of the talcum powder samples are placed on two glass slides, a drop of the 1.605 refractive index fluid was placed on each of the three . . . mounts, stirred with the point of a scalpel blade, and then covered with . . . [a] glass cover slip."⁷¹ This procedure is shown in **Figure 19**. In this method, grains with exactly the same n will be invisible, while grains with higher or lower values of n will appear to have positive or negative relief.

[Figure 19 on next page.]

⁷⁰ 2/1/19 MDL Report, page 5.

⁷¹ 2/1/19 Longo/Rigler MDL Report, page 10.

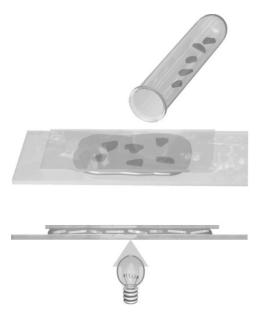


Figure 19. For microscopy using refractive index oils, grains of mineral are placed on a microscope slide surrounded by a liquid with a refractive index matching the mineral. From Dyar and Gunter (2008), Figure 17.6.

In the technique called "dispersion staining" used by Drs. Longo and Rigler, a special $10 \times$ objective is used on the microscope to cause dispersion of light waves and create certain specific colors. The colors become more red, orange and yellow when $n_{\rm mineral} > n_{\rm oil}$, and more like indigo, purple and turquoise when $n_{\rm mineral} < n_{\rm oil}$. The color will change when the microscope stage is rotated because the densities of atoms are different along different planes in each crystal's structure.

The problems that arise from the optical images and their interpretation are similar to those of SAED. It is difficult to distinguish minerals with very similar structures (e.g., tremolite and actinolite) because the spacings of layers of atoms and the compositions are identical or very similar. As a result, many of the minerals from **Table 1**, as well as many other amphibole group minerals, will have overlapping optical characteristics that can make them difficult to distinguish from one another, rendering any optical analyses (even PLM, even using dispersion staining) inconclusive.

Consider, for example, the list of mineral refractive indices at this url: https://nature.berkeley.edu/classes/eps2/wisc/ri.html, or the extensive list of Shannon et al. (2017). A survey of the Berkeley list shows that 87 of the 204 mineral species (43%) listed there have at least one refractive index that falls in the range of n = 1.55-1.65. Careful PLM work requires that refractive indices be obtained at different orientations (preferably using a range of oils to pinpoint the value of n at each orientation) to refine the identification. It may be necessary in geological studies to corroborate microscope mineral identifications using chemical data obtained by a high-accuracy method.

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Shannon et al. (2017) Refractive indices of minerals and synthetic compounds. *Amer. Mineral.*, **102**, 1906-1914. The refractive index table is available under the listing for page 1906 on this url: http://www.minsocam.org/MSA/AmMin/TOC/2017/Sep2017_data/Sep2017_data.html.

B. All Of The ISO PLM Tests Returned Results Consistent With A Finding Of No Asbestos

There are three sources of ISO PLM data: (1) results from the analysts working for Drs. Longo and Rigler; (2) results from the J3 laboratory that are reported in the Longo/Rigler MDL Reports; and (3) results from the J3 laboratory that (for some unknown reason) are not reported in the Longo/Rigler MDL Reports. The Longo/Rigler MDL Reports do not synthesize these data in one place. Notwithstanding that issue, the results appear to be as follows:

- All the reported ISO PLM test results whether from Drs. Longo and Rigler's laboratory or the J3 laboratory were either "no asbestos detected" or less than 0.1% by weight asbestos detected, which is consistent with detecting no asbestos.
- Of the at least 39 bottles tested by J3 using ISO PLM, the result of each was "no asbestos detected." ⁷³
- There are at least eight instances of, for a single bottle, J3 finding "no asbestos detected," while Drs. Longo and Rigler's analysts found "< 0.1% by weight" of asbestos by ISO PLM, ⁷⁴ as follows:

MDL SAMPLE	LONGO/RIGLER ISO PLM RESULT (wt%)	J3 RESOURCES ISO PLM RESULT (wt%)
M68503-004	< 0.1 Trem/Act	No asbestos detected
M68503-026	< 0.1 Trem/Act	No asbestos detected
M68503-023	< 0.1 Anth	No asbestos detected
M68503-042	< 0.1 Trem/Act, < 0.1 Anth	No asbestos detected
M68503-057	< 0.1 Trem/Act, < 0.1 Anth	No asbestos detected
M68503-020	< 0.1 Anth	No asbestos detected
M68503-059	< 0.1 Trem/Act, < 0.1 Anth	No asbestos detected

Thus, in summary, **all** the ISO PLM testing was consistent with the **absence of asbestos** in the samples.

Moreover, there are also 10 instances where the Longo/Rigler reports identify concentrations of asbestos by the "Blount PLM" method that, if correct, are well above the sensitivity limits of ISO PLM. Thus, **all** such "positives" should have registered as positives on the ISO PLM. Yet here are the results, comparing these 10 instances to the results from the PLM testing at J3:

These are Nos. M68503-010, M68503-009, M68503-024, M68503-004, M68503-014, M68503-011, M68503-027, M68503-019, M68503-038, M68503-026, M68503-005, M68503-029, M68503-021, M68503-023, M68503-028, 02D, M68503-046, M68503-042, M68503-057, M68503-020, 07D, 15D, 50D, M68503-059, 10D, 38D, 63D, 52D, 65D, 37D, 45D, 51D, 66D, 21D, M68503-001, 31F, 31G, M68503-016 and M68503-01.

These are Nos. M68503-004, M68503-026, M68503-023, M68503-042, M68503-057, M68503-020, M68503-059 and M68503-001.

LONGO/RIGLER	LONGO/RIGLER BLOUNT	J3 RESOURCES ISO PLM
SAMPLE	PLM RESULT (wt%)	RESULT (wt%)
O7D STS	0.2 Trem/Act, 0.5 Anth	No asbestos detected
15D STS	0.3 Trem	No asbestos detected
10D STS	0.2 Trem/Act	No asbestos detected
38D STS	0.2 Trem/Act	No asbestos detected
63D STS	0.2 Trem/Act	No asbestos detected
52D STS	0.2 Trem/Act, 0.5 Anth	No asbestos detected
65D STS	0.2 Trem/Act, 0.2 Anth	No asbestos detected
66 D STS	0.1 Trem/Act	No asbestos detected
31F STS	0.3 Trem/Act, < 0.1 Anth	No asbestos detected
31G STS	0.7 Tre/Act	No asbestos detected

Thus, even where the Longo/Rigler Blount PLM testing result purportedly was seven times the sensitivity reported for the ISO PLM testing, it did not register on the latter. This represents a gross inconsistency, and suggests issues with the PLM work at either or both of Longo/Rigler's laboratory and J3. These results show that the PLM work in the Longo/Rigler MDL Reports does not appear to be reproducible.

C. **Dispersion Staining Was Not Used Correctly**

The Longo/Rigler MDL Reports purport to find asbestos using dispersion staining, but generally accepted scientific methodologies were not followed. The goal of this methodology is to "meet the requirements set forth by EPA (1993) and NIST (1994) [...in which] analysts must measure and record two principle refractive indices, n_{α} and n_{γ} , of every type of asbestos mineral found in bulk samples"75

The protocol described by the Su article, which Dr. Longo testified that his analysts followed, ⁷⁶ sets forth a particular methodology to do the dispersion staining characterization described above. It recommends employing a statistical observation method to determine the refractive index. This involves the following:

> "To estimate n_{α} , analysts randomly select a fiber, rotate it to the extinction position when the fiber elongation axis is nearly parallel (tremolite or actinolite) or exactly parallel (anthophyllite) to the N-S cross hair (assuming the vibration direction of polarizer is E-W) and observe its CSDS color. After 10 to 20 fibers are examined in this way, the fiber with the longest 10 is assumed to exhibit the RI value closest to n_{α} . To estimate n_{γ} , 10 to 20 fibers are examined at the extinction position when the fiber elongation axis is nearly parallel (tremolite or actinolite) or exactly parallel (anthophyllite) to

2/5/19 Longo Depo. 285:9-14.

⁷⁵ Su (2003).

the E-W cross hair; the fiber with the shortest 10 is then assumed to exhibit the RI value closest to n_{γ} ."

There is no indication on any of the PLM outputs in the Longo/Rigler MDL Reports that multiple grains were measured, as recommended in the protocol.

Moreover, **Figure 20** shows an image from the Longo/Rigler MDL Report of a field of talc particles with one amphibole crystal that is a slightly different color than the surrounding talc.

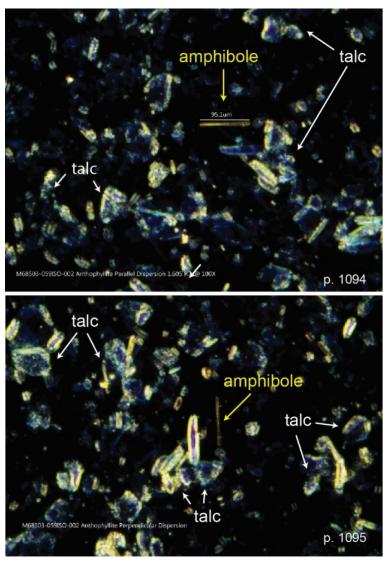


Figure 20. Dispersion staining PLM images showing the same field of view rotated 90°. The change in color of the amphibole grain is diagnostic. Images from pages 1094 and 1095 of the January 2019 Longo/Rigler MDL Report.

Here, the surrounding talc crystals are scattered across the field of view. Some of them appear to have slightly different colors at the edges, but these are likely to be places where the sheets of talc curl up (like chocolate curls). Note that the color variations in these images are very subtle, and it is quite difficult to unequivocally determine if the crystal labelled "amphibole" is

truly a different color than the surrounding grains. This is why the Su (2003) paper recommends looking at many different grains to calculate refractive index correctly.⁷⁷

Another issue is that the dispersion staining images should have used an immersion oil with a refractive index of 1.625 to identify anthophyllite, according to Su (2003) **Table 1**. Instead, the samples were analyzed using a "drop of the 1.605 refractive index fluid." The report cites ISO 22262-1 as the source of the choice of 1.605, when in fact, the report states "For identification of tremolite, actinolite, anthophyllite, and richterite/winchite, RI liquids in the range 1.605 to 1.660 are required, at intervals of 0.005." There is no evidence in the Longo/Rigler MDL Reports to indicate that such a progression of oils was used.

A second example of a confusing PLM image is given in Figure 21.

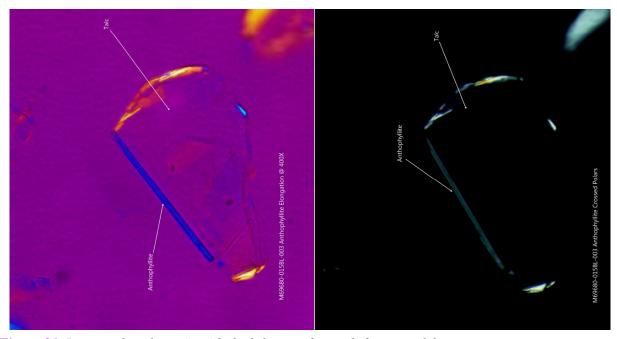


Figure 21. Images of a talc grain with the leftmost edge curled up out of the page.

The curled edge is erroneously identified in the Longo/Rigler MDL Reports as a separate crystal of anthophyllite, but this is highly unlikely. The "anthophyllite" grain would not just "happen" to lie exactly along the edge of the talc grain.

The view at left is pink because it is a dispersion staining image, in which a special wave plate is inserted in the microscope to make the colors more intense (and more diagnostic). These are images of a grain of talc. The straight edge of the talc grain is labeled as "anthophyllite," but in fact, this is a curled-up edge of the talc grain. Because of the curl, the grain is showing a different orientation within the crystal structure, which has different optical properties, and therefore shows

Su (2003) at 1980 ("After 10-20 fibers are examined in this way. ...10-20 fibers are examined at the extinction position...").

⁷⁸ 1/16/19 Longo/Rigler MDL Report, page 9.

⁷⁹ 1/16/19 Longo/Rigler MDL Report, page 15.

up as blue. Very few of the PLM images in the Longo/Rigler MDL Reports have labels. There is little discussion of PLM results in any of the reports.

There is also no mention of utilizing proper protocol for distinguishing between talc and anthophyllite with dispersion staining. As noted in ISO 22262-1:

"Some sources of talc contain fibres that can be mistaken for anthophyllite. These fibres have intergrowths of both the anthophyllite and talc crystal structures. The fibres exhibit refractive indices that are lower than those of anthophyllite and intermediate between those of talc and anthophyllite. If this type of fibre is present, examine the sample in a liquid of RI 1,615. If no γ indices are observed that are higher than 1,615, classify the fibres as talc. Classify any fibres with γ indices equal to or exceeding 1,615 as anthophyllite."

It does not appear that this protocol was followed.

D. The "Point Counting" Method Was Not Used, Yet It Is The Only Way To Estimate Concentration By PLM, As Recognized By ISO 22262

Summary: The ISO 22262-1 protocol provides a methodology for estimating concentration by PLM using "point counting." Drs. Longo and Rigler and their analysts did not do this. Instead, their estimates were based on purported charts generated by Drs. Longo and Rigler's lab's own methodology that are not accepted by the scientific community and that were not produced.

The standard method for estimating concentrations of minerals in geological materials by PLM is called "point counting." In this technique, a sliver of rock or loose particles is placed on an *x-y* stage on a microscope so it can be moved in different directions. The operator then looks down the microscope and uses the technique of choice (usually the optical properties of the minerals as they interact with plane polarized light) to identify the grain at the center of the field of view. Then the stage is moved slightly and the process is repeated. Typically, 100-400 locations on a rock are thus analyzed, from which a quantitative estimate of the abundance of minerals in the rock can be obtained. It is well-recognized that the accuracy of a point count depends on the number of grains counted. This is acknowledged in ISO 22262-1, which says:

"The statistical reliability of a point count for determination of asbestos depends on the number of asbestos points, not on the total non-empty points examined. Accordingly for comparison against a regulatory standard of 0.1 % asbestos by weight, continue point

-

An excellent summary of this method is given at http://geologylearn.blogspot.com/2015/08/point-counting-technique-used-in.html. *See also* Neilson and Brockman (1977) *Amer. Mineral.*, **62**, 1238-1244.

counting until either a minimum of 20 asbestos points, or the equivalent of 13,000 non-empty points have been accumulated."81

At his deposition, Dr. Longo confirmed that the Longo/Rigler MDL Reports did not use point counting. Rather, the Longo/Rigler MDL Reports state that "a visual estimation of the quantity of asbestos observed was based on eye calibration through review of lab generated weight percent standards." First off, Drs. Longo and Rigler did not produce those "lab generated weight percent standards," so there is no evidence that the standards are reliable or based in accepted science. Moreover, given the similarities in color of talc and amphibole in these images, these visual estimates are unreliable and unreproducible. The estimates of abundances are orders of magnitude different from the estimates produced by the TEM measurements due to analytical sensitivity issues, also noted by Drs. Longo and Rigler. Probably for this reason, little attention is paid to the amounts of amphibole observed by the PLM method. Instead, PLM is used in the Longo/Rigler MDL Reports mostly to determine presence/absence of amphibole in asbestos. For example, the January 2019 report notes that 20 of the 61 samples tested "were positive for regulated amphibole asbestos."

Moreover, it is not clear what these "weight percentage standards" are, as Drs. Longo and Rigler did not produce them. In that sense, their results are unverifiable and unreproducible.

E. Conclusions Relating To PLM Analysis

- **Dispersion staining was not used correctly.** In contrast to the recommended protocol, the Longo/Rigler MDL Reports use only a single refractive index liquid and do not count the requisite number of particles.
- The Longo/Rigler MDL Reports purport to find asbestos using dispersion staining, but generally accepted scientific methodologies were not followed. Indeed, it does not appear that the protocol cited was followed.
- The reported quantities of asbestos based on PLM used "visual estimation" rather than point counting. Apparently, the estimates were produced by comparisons against unspecified and unregulated "weight percentage standards." The results are very different from those obtained by TEM, and are mostly ambiguous.
- Drs. Longo and Rigler and their analysts did not follow the methodology for estimating concentration by PLM using "point counting." Instead, their estimates were based on purported charts generated by Drs. Longo and Rigler's lab's own methodology that are not accepted by the scientific community and that were not produced.

ISO 22262-1, page 29.

⁸² 2/5/19 Longo Depo. 269: 20-23, 272:15-17.

⁸³ 1/16/19 Longo/Rigler MDL Report, page 9.

⁸⁴ 1/16/19 Longo/Rigler MDL Report, page 7.

VII. MORPHOLOGY IDENTIFICATION USING VISUAL INSPECTION BY TEM

The primary method used in the Longo/Rigler MDL Reports to establish having found asbestiform habit minerals is visual inspection using TEM images. Yet, the methods that they purport to follow expressly state that TEM "cannot be used to discriminate between individual fibers of the asbestos and non-asbestos analogs of the same amphibole material." 85

Evaluation of whether a particle is a bundle, fiber or cleavage fragment by visual inspection is highly subjective and subject to personal biases, as will be shown below. This section addresses how the methodologies in the Longo/Rigler MDL Reports for identifying talc are scientifically unacceptable, and that the conclusions reached thereby are unsupportable.

A. Differences Among Fibers, Bundles And Fragments

Summary: There are important morphological differences between asbestiform fibers, asbestiform bundles and non-asbestiform cleavage fragments.

Fibers are described as very thin fibrils, usually less than 0.5 mm in width (*cf.* **Figure 1**), rather than the chunky particles identified in the Longo/Rigler MDL Reports. Fibers are identified on the basis of their curvature, flexibility and high tensile strength. **Figure 22** below shows the visual difference between asbestiform and non-asbestiform versions of the same minerals:

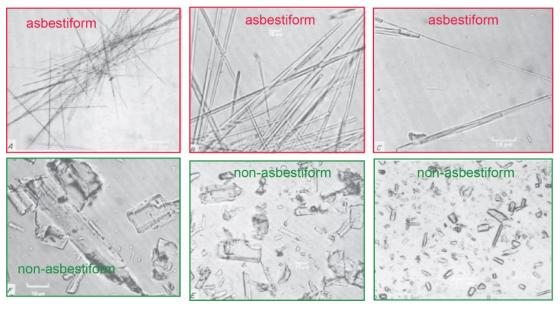


Figure 22. Examples of asbestiform vs. non-asbestiform particles from Figure 39 of the classic paper by Campbell et al. (1977).

The differences between asbestiform and non-asbestiform minerals go back to the first definitions given in this report. Recall that the ISO 22262-1, 2, 3 documents define asbestiform as a "specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength

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ISO 10312 (1995), at 1; *see* ISO 13794 (1999) (same), at 1; ISO 22262-1 (2012) at 31 ("Full details relating to identification of asbestos fibers using TEM are given in ISO 10312 and ISO 13794.").

and flexibility." The particles in the top row of **Figure 22** all seem to comply with this definition. A non-asbestiform particle is often called a cleavage fragment, which means it is a small particle that has broken off a larger crystal. These will appear to be chunkier, as seen in the bottom row of **Figure 22**.

Even if a particle of a mineral that can have an asbestiform habit is long and thin, it may not be an asbestos fiber. Again, that term relates as much to physical properties such as tensile strength and flexibility as it does to aspect ratio. **Figure 23a** shows three examples of fibers that are not "asbestos," but rather cleavage fragments of non-asbestiform amphibole.

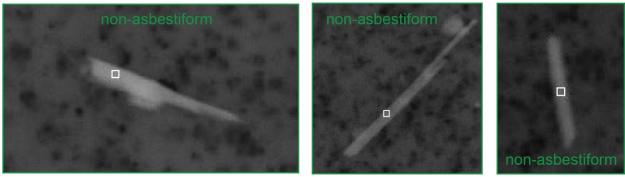


Figure 23a. Images of non-asbestiform particles from Gunter (2010).

Asbestos particles can come in fibers or bundles. Bundles occur as separable groups of parallel fibers with **splayed** ends and matted masses as seen in **Figure 23b**. They are defined in ISO 22262-1 as structures "composed of parallel, smaller diameter fibers attached along their lengths."



Figure 23b. Image of tremolite bundle of asbestiform particles from Harper et al. (2010).86

Harper et al. (2014) Characterization of Lone Pine, California tremolite asbestos and preparation of research material. *Ann. Occp. Hyg.*, **2014**, 1-13.

The assessment of whether a particle is an asbestiform fiber or bundle versus a cleavage fragment is fairly straightforward. Asbestiform fibers are greatly elongated with high aspect ratios. At the end of a bundle, the fibers generally bend out like the straws at the end of a broom. Note in **Figure 23b** that the particles do splay out at the end, and it is obvious that the bundle at lower right is indeed "composed of parallel, smaller diameter fibers attached along their lengths."

Alternatively, cleavage fragments tend to be short and fat particles that cannot maintain the thinness and long aspect-ratios associated with asbestiform minerals because they are generally brittle. Non-asbestiform cleavage fragments are blocky, and may show stair-step cleavage fractures on the ends of the grains.

B. Drs. Longo And Rigler Misidentify Cleavage Fragments As Bundles And Fibers

Summary: Drs. Longo and Rigler and their analysts did not apply objective criteria for distinguishing between cleavage fragments, bundles and fibers, as demonstrated by the inconsistent results of their analysts.

The Longo/Rigler MDL Reports consistently demonstrate confusion over the identification of non-asbestiform cleavage fragments vs. the asbestiform habit. For example, the particles shown in **Figure 23c**, which are explicitly identified as fibers and bundles, are effectively identical to the non-asbestiform cleavage fragments in the Longo/Rigler MDL Reports shown in **Figure 23d**.

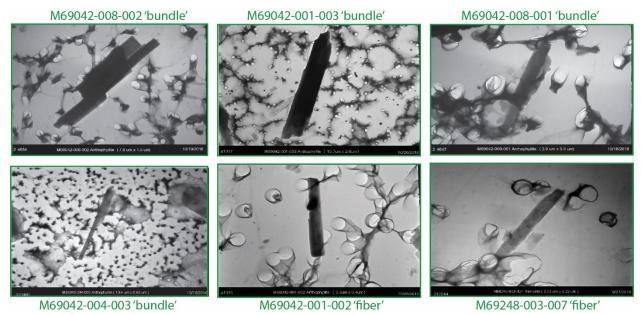


Figure 23c. Particles from the 1/16/19 Longo/Rigler MDL Report and assignments. Compare to Figures 23a, b and c. This comparison shows visually that the bundles and fibers identified in the Longo/Rigler MDL Report are NOT fibers and bundles, but rather, non-asbestiform particles.



Figure 23d. Dispersion staining photomicrographs from the January 2019 Longo/Rigler MDL report with ambiguous annotations. For sample M69503-010, the PLM ANALYSIS report states the presence of "actinolite/tremolite cleavage fragments" (p. 120).

For sample M69503-026, the PLM ANALYSIS report states the presence of "tremolite/actinolite asbestos observed. Actinolite/Tremolite cleavage fragments observed."

Compare these images to **Figure 23e**, which shows PLM dispersion staining images of tremolite cleavage fragments from the peer-reviewed paper by Pierce et al. (2017). These dispersion photomicrographs were imaged by RJ Lee Group and Forensic Analytical, Hayward, CA. Particles in **Figures 23e** and **23f** are effectively identical, and here, the analysts employed by Drs. Longo and Rigler correctly identify these particles as cleavage fragments.

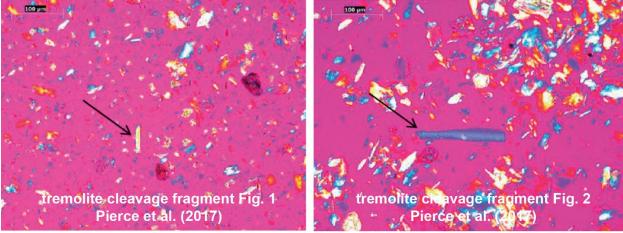


Figure 23e. PLM dispersion staining images of tremolite non-asbestiform cleavage fragments from the peer-reviewed paper by Pierce et al. (2017).

But the particles seen in **Figure 23f**, which are strikingly similar, are indicated in the Longo/Rigler MDL Reports to be **asbestiform bundles**, even though they demonstrate none of the objective criteria for such bundles. **They look nothing like the bundle shown in Figure 23c.**





Figure 23f (left) The TEM report for sample M69503-010 (p. 131) identified 4 bundles of tremolite. No mention is made of cleavage fragments (right).

The TEM report for sample M69503-026 (p. 321) identified 31 bundles of tremolite. Note that the shape of the "bundles" identified in the Longo/Rigler TEM images is the same as the shape of the cleavage fragments in Pierce et al. (2017).

There is obvious confusion in the Longo/Rigler MDL Reports over these identifications. Nearly all of the amphiboles referred to as "fibers" and "bundles" in the Longo/Rigler MDL Reports are likely to be either non-asbestiform amphibole cleavage fragments, or in some cases, talc particles viewed on edge.

C. Results Of Analysts Working For Drs. Longo And Rigler Are Inconsistent

Summary: The results reported in the Longo/Rigler MDL Reports are inconsistent from analyst to analyst, highlighting the unreliability of their method of visual identification using TEM.

The inconsistent results of the analysts working for Drs. Longo and Rigler in characterizing fibers and bundles is further proof of the lack of repeatability of the analysis in the Longo/Rigler MDL Reports. Because criteria for describing particle morphology are so ambiguous, it comes as no surprise that different analysts would produce wildly variable results (**Figure 24**). Some analysts, such as Mehrdad Motamedi, are far more likely to classify a particle as a fiber, and Anthony Keaton, for example, finds far more fibers than either Jayme Callan or Lee Poye.

[Figure 24 on next page.]

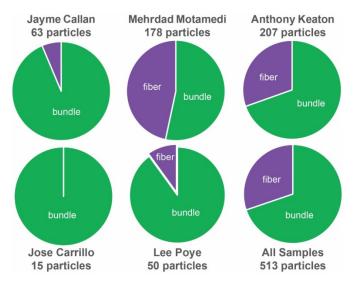


Figure 24. Comparison of fiber vs. bundle identifications made by different analysts. Note that totals are different from those in the mineral identification figures because morphologies were not reported for some particles. Data sources as in Figure 8.

Figure 25 shows that the percentage of observed bundles vs. fibers also increases over time. Because the talc samples studied were randomly selected from various collections, such a conclusion is highly unlikely, and simply shows operator bias. This renders the distinctions made in the Longo/Rigler MDL Reports between fibers and bundles arbitrary.

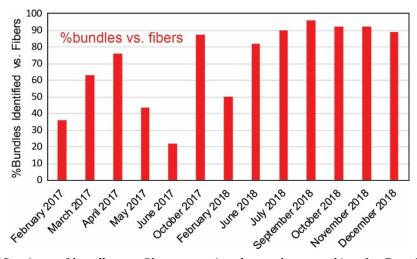


Figure 25. Identifications of bundles vs. fibers over time by analysts working for Drs. Longo and Rigler. Data sources as in Figure 8.

Finally, **Figure 26** shows results from a test undertaken to supposedly demonstrate the "Coefficient of Variation" of their analysts.⁸⁷ The stated goal of this document is "to determine the MAS [Longo/Rigler] TEM analysis coefficient of variation (CV or relative standard deviation RSD)" of measurements made on cosmetic talc spiked with asbestos standard reference material. In other words, this testing sought to confirm consistency of results from the company's testing.

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TEM Coefficient of Variation for Tremolite and Anthophyllite in Talc: A Quality Control Study.

Two TEM grids were studied. Supposed "asbestos" particles were searched for in each of 100 squares in each grid. Then four of the analysts working for Drs. Longo and Rigler were asked to measure the particles and judge if they are fibers or bundles. Each analyst looked at exactly the same particles. The document does calculate the mean and standard deviation of the **number** of particles observed on each sample. Standard deviation in this instance indicates how reproducible the measurement is, but says nothing about accuracy – i.e., how close it is to the true value.

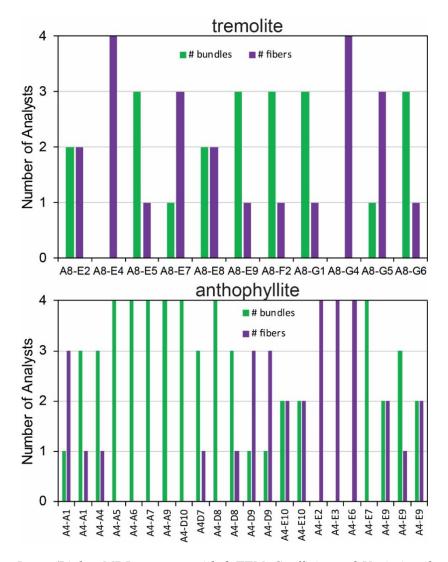


Figure 26. The Longo/Rigler MDL report entitled TEM Coefficient of Variation for Tremolite and Anthophyllite in Talc: A Quality Control Study shows results from four different analysts who identified the same particles in the same set of two TEM grids, one for tremolite (top) and one for anthophyllite (bottom). Out of 44 particles, there are only 9 cases in which all four analysts agree on whether a particle is a fiber or a bundle. In other words, they disagree 80% of the time. This represents an unacceptable inconsistency and reveals the arbitrary nature of these judgments.

But the document also shows the alarming inconsistency of analysts working for Drs. Longo and Rigler with respect to identification of bundles vs. fragments. **Figure 26** shows that the four analysts all agreed on fiber vs. bundle identification for only 9 of the 44 particles. In other words, only 20% of the time did the four analysts concur on their judgments. This result, when

coupled with the results presented in **Figures 24** and **25**, firmly establishes that the visual distinctions made between particles and bundles by Drs. Longo and Rigler's analysts are scientifically invalid and unacceptable.

D. Statistical Distinctions Between Fibers And Fragments

As noted above, visual distinctions between fibers, bundles of fibers and non-asbestiform cleavage fragments are quite subjective – and in the case of the analysts employed by Drs. Longo and Rigler, done unreliably and in a way that is not reproducible. There is a scientifically accepted methodology, however, for determining whether a population of amphibole is asbestiform or non-asbestiform, based on their **statistical** distinction between their distributions of sizes.

Specifically, a population of amphiboles will have different, characteristic distributions of aspect ratio, depending on whether they are asbestiform or non-asbestiform. ⁸⁸ These can be analyzed accurately and in a way that is reproducible using statistics.

1. Background on statistical tests of population differences

Summary: A *t* test assesses whether two populations have the same or different means. We can use this test to determine whether the data from the Longo/Rigler MDL Reports show a population of non-asbestiform cleavage fragments or asbestiform fibers.

As noted in my geostatistics textbook, "today's earth scientists need to be able to critically evaluate sampling designs, to understand the concept of statistical analysis, and be able to evaluate and interpret the results of statistical tests applied in a wide range of fields." In considering the problem of particle size distributions in talcum powder impurities, only the most basic of such statistical methods is necessary – the need to distinguish among the average particle dimensions based on the ratio of length to width (aspect ratio). Comparisons between the mean (\approx average) are so basic that even Microsoft Excel can calculate them. Here, the need for statistics is based on the problem of discriminating among known populations of non-asbestiform tremolite, asbestiform tremolite and the particle measurements in the Longo/Rigler MDL reports. For simplicity, these populations can be compared two at a time.

The appropriate test here is called a **two-tailed** *t* **test**. It allows for testing of the hypothesis that "the two samples are drawn from populations with different means." In conducting this analysis, the underlying assumption is that the two populations (here, asbestiform vs. non-

^{2/5/19} Longo Depo. 290:11-21 ("Q. Okay. And the nonasbestiform version of the same amphibole has a different characteristic distribution? A. Yes, it does. Q. And you did not generate a particle size distribution chart like the one in Blount's paper -- the ones in Blount's paper in your report? A. Not for the MDL samples, no."); 2/6/19 Rigler Depo. 190:10-25 ("Q. That there's a characteristic -- there is a characteristic particle size distribution for asbestos; is that correct? A. Well, depending on how that sample's been processed, you're going to have different fiber sizes, different – they're going to be different. You're going to have different aspect ratios and different sizes. Q. For any given sample that everyone agrees is asbestos, it's going to have a characteristic particle size distribution; right? A. It can."); *id.* 191:10-13 ("Q. Do cleavage fragments have a different particle size distribution than asbestos? A. They can.").

McKibbin and Dyar (201) *Geostatistics Explained*. Cambridge University Press.

⁹⁰ *ibid*.

asbestiform) have comparable variation – this is called "equal variance" in statistics-speak. Then a critical value for the *t* test is selected to specify the desired level of confidence for the answer. It is conventional to use a 95% confidence limit, which means that conclusions can be drawn with a 95% chance that they are correct.

Thus, Figure 27a shows a normal distribution for a hypothetical population.

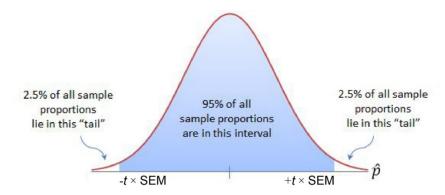


Figure 27a. Schematic of a normal population distribution illustrating the concept of confidence interval.

Here, the 95% confidence interval, which is estimated from a sample by using the *t* statistic, is indicated in blue, where SEM is the standard error of the mean of the population. ⁹¹ Therefore, 5% of the means of this sample will lie outside this range, but if the mean lies within the 95 interval, then it will be considered to have come from a population with a mean the same as this one.

The *t* test is used to assess whether the mean of a sample lies within or outside the 95% confidence limit of a population, implying that the two samples come from the same population. **Figure 27b** shows two populations that are being compared:

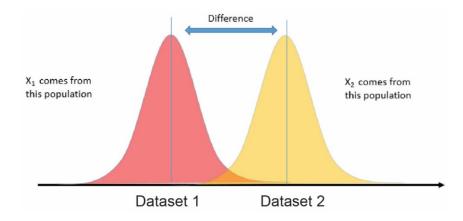


Figure 27b. Adapted from https://rexplorations.wordpress.com/2015/08/13/hypothesis-tests-2-sample-tests-ab-tests/.

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⁹¹ McKillip and Dyar (2010) *Geostatistics Explained*. Cambridge University Press.

In the figure, the two populations barely overlap, and only within the 2.5% confidence limit on the extreme edges of the curves. As illustrated, these two populations can be said to derive from different sources.

A *t* test assesses whether two populations have the same or different means, which can be employed to assess if the data from the Longo/Rigler MDL Reports come from a population of non-asbestiform cleavage fragments or asbestiform fibers.

2. Statistically significant differences are seen between asbestiform tremolite and non-asbestiform tremolite cleavage fragments

Summary: Use of the *t* test applied to peer-reviewed data shows two distinct populations of shapes: asbestiform and non-asbestiform.

Now, consider the application of the two-tailed *t* test to making distinctions between asbestiform and non-asbestiform particle morphologies. Here, the hypothesis is whether the aspect ratio (particle length divided by width) is statistically different for non-asbestiform tremolite than for asbestiform tremolite. To understand this problem, comparisons among pairs of datasets can be made using three available populations of data:

- 1. Non-asbestiform amphiboles from the compilation of **Wylie and Virta (2016)**, who provide data on the lengths and widths of amosite, crocidolite, chrysotile and nonfibrous tremolite;⁹²
- 2. Asbestiform tremolite data from **Harper et al. (2014)**;⁹³ and
- 3. Particles in the **Longo/Rigler MDL Report**.

Using these data, the first test is whether asbestiform and non-asbestiform particle populations are different. These are shown here, in **Figure 28a**:

[Figure 28a on next page.]

Wylie and Virta (2016) Size distribution measurements of amosite, crocidolite, chrysotile, and nonfibrous tremolite. *Digital Repository at the University of Maryland*, http://dx.doi.org/10.13016/M2798Z.

Harper et al. (2014) Characterization of Lone Pine, California tremolite asbestos and preparation of research material. *Ann. of Occp. Hyg.*, **2014**, 1-13, https://academic.oup.com/annweh/article/59/1/91/2464346#supplementary-data.

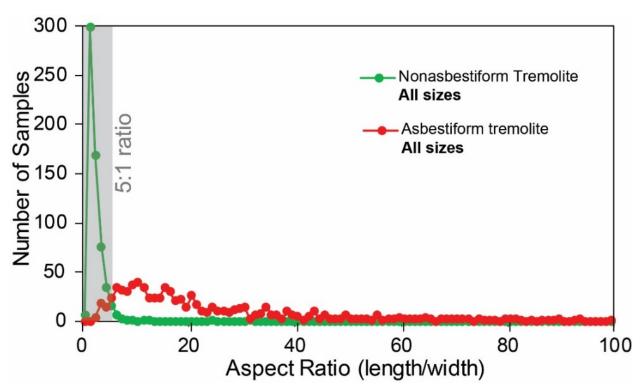


Figure 28a. Distribution of aspect ratios for non-asbestiform vs. asbestiform particles of amphibole, the dominant impurity found in talcum powder. This graph shows all the data in the two repositories, Wylie and Virta (2016) and Harper et al. (2014).

Using the available peer-reviewed data, cleavage fragments (non-asbestiform tremolite) are indicated in green and asbestiform tremolite in red. Note that asbestiform fibers can have extremely large aspect ratios – the longest particle in the asbestos suite is $55 \, \mu m \times 0.15 \, \mu m$ – it is so large that it has been cropped out of the figure for clarity. In fact, there are 13 samples in the asbestiform data set with aspect ratios over 100. By contrast, of the 615 non-asbestiform tremolite cleavage fragments, only one has an aspect ratio greater than 32 (it has a value of 115.38). In other words, the two types of particles have very different characteristics. Note also the shaded bar, which indicates the region with an aspect ratio of 5:1 or less. Most of the non-asbestiform tremolite is within that region, most of the asbestiform tremolite is outside.

Figure 28b is the exact same graph as **Figure 28a**, just with the "less than 5:1" region deleted, which becomes interesting when discussing the population distributions. Again notice that there are many particles even at very high aspect ratios above 10 – this is a characteristic of asbestiform fibers. In comparison, almost none of the non-asbestiform particles has an aspect ratio above 10.

[Figure 28b on next page.]

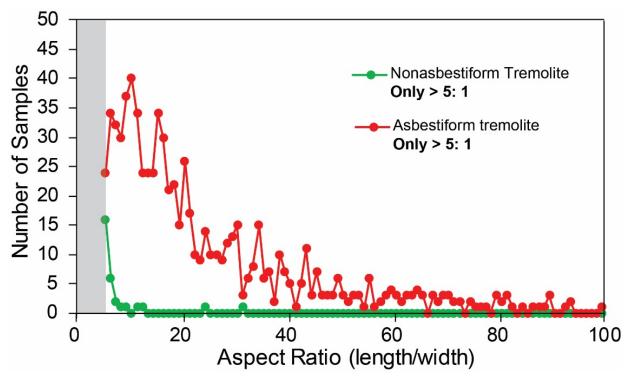


Figure 28b. The same data as Figure 28a, except with the "5:1 and less" region deleted.

Visual examination of these plots suggests that non-asbestiform tremolite has different shapes than asbestiform tremolite. This can be proven using the statistical *t* test described above to see if these samples come from the same population. **Figure 27a** showed the distribution curve for ONE population – but now the test asks if the asbestiform population is the same as the non-asbestiform population. In other words, do the two population distribution curves overlap?

To make this comparison rigorously, the t test described above is used to compare the means of these two populations. The results shown below in Table 3 demonstrate that these two populations are statistically different. The top half of the table compares all the population data for asbestiform vs. non-asbestiform tremolite – some 615 and 817 samples, respectively. There, the value of the t statistic is -21.44, a value that is far outside the 95% confidence limit. This means that the non-asbestiform and asbestiform tremolite particles really are drawn from samples with different means.

[Table 3 on next page.]

Table 3. Results of *t*-Tests Assuming Equal Variance

ALL Non-asbestiform tremolite (NAT) compared to asbestiform tremolite (AT)

	NAT	AT
Mean	2.70	25.73
# Observations	615	817
t Stat	-21.44	
t Critical two-tail	1.96	

-21.44 lies outside the 95% confidence interval, so the means are different and these samples are not drawn from the same population.

Non-asbestiform tremolite (NAT) compared to asbestiform tremolite (AT) with aspect ratios >5:1

	NAT	AT
Mean	10.76	26.77
# Observations	35	780
t Stat	-3.54	
t Critical two-tail	1.96	

-3.54 lies outside the 95% confidence interval, so the means are different and these samples are not drawn from the same population.

3. Comparison to results in the Longo/Rigler MDL Reports

Summary: Use of the *t* test applied to data from the Longo/Rigler MDL Reports vs. published population data shows that the particles in talcum powder belong to the population of non-asbestiform amphibole.

Returning to the visual evidence presented in **Figures 28a and b**, now consider **Figure 28c**, which is **Figure 28b** with results from the Longo/Rigler MDL Report added in black:

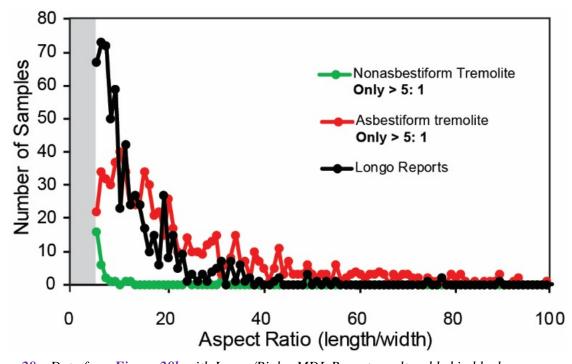


Figure 28c. Data from Figure 28b with Longo/Rigler MDL Report results added in black.

Significantly, the data from the Longo/Rigler MDL Reports appears to be trending toward a peak aspect ratio below 5:1, which is characteristic of non-asbestiform tremolite.

Unfortunately, the data collected by the analysts working for Drs. Longo and Rigler do not represent the population of non-asbestiform particles because **they only counted particles with aspect ratios of greater than 5:1.** 94 Their experimental design neglected to count the vast majority of particles in the typical size range for cleavage fragments, as seen in **Figure 28b**. This omission renders their analysis completely invalid. Indeed, this is a rejection of ISO 22262 methodologies, in particular as found on page 23 of ISO 22262-1, which makes certain observations about whether a population can be considered asbestiform based on observations of the **entire** population, but certainly not the population cut off at a 5:1 aspect ratio.

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The methodologically recognized dimensions for fibers are 3:1 for polarizing light microscopes (PLM) and 5:1 for TEM measurements. Although the Longo/Rigler MDL Reports utilize PLM for evaluating optical properties, the reports do not give aspect ratios for studied particles, either on the photomicrographs themselves or in any tables.

Particles measured in the Longo/Rigler MDL Reports compared to asbestiform tremolite (AT) with aspect rations >5:1

	\mathbf{AT}	LONGO/RIGLER
Mean	26.77	13.34
# Observations	780	632
t Stat	12.09	
t Critical two-tail	1.96	

12.09 lies outside the 95% confidence interval, so the means are different and these samples are not drawn from the same population.

Particles measured in the Longo/Rigler MDL Reports compared to non-asbestiform tremolite (NAT) with aspect ratios >5:1

	NAT	LONGO/RIGLER
Mean	10.76	13.34
# Observations	35	632
t Stat	-1.41	
t Critical two-tail	1.96	

-1.41 lies inside the 95% confidence interval, so the means are the same; samples are drawn from the same population.

Fortunately, the Wylie and Virta (2016) data set is large enough to include a significant number of samples with aspect ratios >5:1, so comparisons with that limited size range can still be made. To make this conclusion using statistics, **Table 4** shows results from tests comparing data from the Drs. Longo and Rigler's MDL Reports for particles with aspect ratios >5:1 against data from Wylie and Virta (2016) and Harper et al. (2014). The top set of data compares asbestiform particle aspect ratios against data from Drs. Longo and Rigler's MDL Reports. Here, the t statistic of 10.68 lies outside the value of 1.96 for the 95% confidence limit. This establishes that particles measured by Drs. Longo and Rigler do not belong to the same populations as those of asbestiform tremolite on the basis of their shapes. In other words, the **asbestiform and non-asbestiform populations are so different** that they can be distinguished even when a flawed experimental design provides data on particles with aspect ratios greater than 5:1. **In summary, the data from the Longo/Rigler MDL Reports show that even though counts and measurements are only reported for particles with aspect ratios >5:1, statistical tests show that they have identified a population of non-asbestiform cleavage fragments in the talcum powder tested.**

Note that the available morphology data for asbestiform and non-asbestiform particles come from analyses of the mineral tremolite, which is the dominant species identified by Drs. Longo and Rigler in their November 2018 MDL Report (67%). Most, if not all, of the anthophyllite particles identified by Drs. Longo and Rigler are likely to be talc, and there is no relevant data set for talc.

Wylie and Virta (2016) Size distribution measurements of amosite, crocidolite, chrysotile, and nonfibrous tremolite. *Digital Repository at the University of Maryland*, http://dx.doi.org/10.13016/M2798Z.

Harper et al. (2014) Characterization of Lone Pine, California tremolite asbestos and preparation of research material. *Ann. of Occp. Hyg.*, **2014**, 1-13, https://academic.oup.com/annweh/article/59/1/91/2464346#supplementary-data.

E. Summary Of Particle Morphology Conclusions

In summary, the following flaws in evaluating particle morphology render the Longo/Rigler MDL Reports conclusions invalid:

- Using TEM to visually distinguish non-asbestiform amphiboles and asbestiform amphiboles is scientifically invalid, as stated in the very protocols upon which Drs. Longo and Rigler purport to rely.
- Drs. Longo and Rigler and analysts only counted particles with aspect ratios >5:1 in their TEM studies, which improperly biases their results toward finding an asbestiform particle population.
- Particles in the Longo/Rigler MDL Reports belong to the non-asbestiform tremolite population.
- There is no evidence to suggest that the particles measured by Drs. Longo and Rigler and analysts are asbestos.

VIII. CONCLUSIONS

The core problem with the Longo/Rigler MDL Reports is that the methods utilized in the testing and analyses produce completely inconsistent results, rendering them unscientific and subjective. Indeed, the analysis by Drs. Longo and Rigler presented in the Long/Rigler MDL Reports applied a methodology that was inherently designed to achieve the results they desired for purposes of this litigation, and not scientific.

Despite producing lengthy, descriptive reports and undertaking extensive measurements according to government protocols, Drs. Longo and Rigler did not properly train their personnel for the task of characterizing impurities in talc, where the possible mineralogy is unconstrained. Thus, they lack the background to understand or interpret their results, especially where, as here, the impurities are tiny, and many possible minerals can be present. Personnel employed by Drs. Longo and Rigler lack sufficient knowledge of proper mineralogical terminology beyond the six defined asbestos mineral species, have **no** understanding of the limitations and errors associated with the techniques used, and fail to compare their results to foundation studies in this discipline.

The Longo/Rigler MDL Reports fail to demonstrate an understanding of the accuracy of the Energy Dispersive Spectroscopy (EDS) technique, which is only semi-quantitative with large error bars. The technique is rarely used to determine the chemical composition of minerals in unbounded studies where the possible minerals present cover a large range of possibilities. Within generally accepted error bars on this technique (ignored and unstated by Drs. Longo and Rigler), asbestos minerals are indistinguishable from many of their non-asbestiform counterparts. In particular, EDS cannot differentiate between talc and anthophyllite. It is simply incorrect for Drs. Longo and Rigler to assert that individual amphibole mineral species can be identified by EDS alone.

The Longo/Rigler MDL Reports also use single crystal selected area electron diffraction (SAED) to confirm the EDS mineral identifications based on crystal structure. But the vast majority of the patterns collected by Drs. Longo and Rigler are not indexed, making them very difficult to interpret. Moreover, 25% of all common rock-forming minerals have overlapping similar or identical *d*-spacings to what Drs. Longo and Rigler report. There is no SAED evidence to prove definitively that any specific asbestiform minerals are present.

Polarizing light microscope (PLM) images of various types are used in the Longo/Rigler MDL Reports to prove presence of asbestos in the samples. Again, the PLM images themselves do not uniquely identify "asbestos" mineral species. Rather, some of them can be interpreted to suggest the presence of amphibole group minerals (there are 76 end-members, and many more intermediates) or something else. The latter might be talc, which appears to have very close to the same color as amphibole in the dispersion staining images. The PLM images alone do not categorically prove the presence of any specific mineral species.

The experimental design used by Drs. Longo and Rigler for interpreting the number of non-talc particles present in talcum powder ignores well-known databases on particle dimensions, which indicate that non-asbestiform particles occur with aspect ratios (particle length divided by width) averaging 2.7. They ignore all particles with aspect ratios less than 5:1, rendering the particle dimension analyses nearly useless for distinguishing cleavage fragments from fibers.

Because the distribution of non-asbestiform particles has some samples in the range above 5:1, it is possible to compare that truncated (aspect ratio greater than 5:1) dataset for cleavage fragments (average aspect ratio of 10.8) to that of asbestiform tremolite with ratios above 5 (average ~26.8). The average aspect ratio for all particles measured by Drs. Longo and Rigler is 13.3, suggesting that they are a population of cleavage fragments. Statistical analyses of these aspect ratio populations establish that the particles measured by Drs. Longo and Rigler belong to the population represented by non-asbestiform amphibole.

There is no evidence (EDS, SAED, or otherwise) to confirm that any of the "fibers" identified as anthophyllite by Drs. Longo and Rigler are really anthophyllite.

In summary, the Longo/Rigler MDL Reports contain only subjective analyses with inconsistent results that do not stand up to scientific scrutiny. It appears that Drs. Longo and Rigler and their analysts are just using the equipment and methodology with which they are familiar from their work in bulk asbestos sampling to undertake analyses in a totally different application for which they are inappropriate. The Longo/Rigler MDL Reports are far from the type of quantitative study that would withstand scientific review or be accepted for publication in a peer-reviewed scientific journal. The Longo/Rigler MDL Reports completely fail to demonstrate that any recognized asbestos minerals are present in talcum powder in any concentration with any asbestiform shapes.

Finally, given the compounding nature of all of the individual methodological errors in the Longo/Rigler MDL Reports, any resultant conclusions drawn from testing by Drs. Longo and Rigler and associates are scientifically invalid. Every methodological error in their testing results in false positive findings of asbestos, but not false negatives. The testing described in the reports can only be described as a series of errors, each of which acts to amplify the errors preceding it. The results reported in the Longo/Rigler MDL Reports cannot be considered scientifically valid, calling into question the competence of Drs. Longo and Rigler and their analysts to undertake such work.

MATERIALS REVIEWED AND CONSIDERED

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Global particle table M66514-001

Global particle table M66515-001	
Global particle table M66516-001	

Depositions and Exhibits

am Longo, Nov. 6, 2	2018

Deposition of William Longo, Nov. 27, 2018

Deposition of William Longo, Dec. 5, 2018

Deposition of William Longo, Feb. 5, 2019, and Exhibits

Deposition of Rigler, Feb. 6, 2019, and Exhibits

Deposition of William Longo, Oct. 24, 2018

Deposition of William Longo, Oct. 31, 2018

Deposition of William Longo, Jan. 7, 2019

Scientific Literature

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Campbell et al. (1997) Information Circular 8751, U.S. Bureau of Mines.

Dyar and Gunter (2008) *Mineralogy and Optical Mineralogy*, Mineralogical Society of America

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Miller and Mirtič (2013) Accuracy and precision of EDS analysis for identification of metal-bearing minerals in polished and rough particle samples, *Geologija*, **56**, 5-17.

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Transmission Electron Microscope, 2008 report, Asbestos Analysis Consulting, Newark, DE

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40 C.F.R. 1 (7-1-03 Edition).

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ISO 10312 (1995)

ISO 13794 (1999)

ISO 22262-1 (2012)

ISO 22262-2 (2014)

ISO 22262-3 (2016)

National Institute for Occupational Safety and Health (2011) *Asbestos Fibers and Other Elongate Mineral Particles: State of the Science and Roadmap for Research*, Current Intelligence Bulletin 62

National Research Council (1984) Asbestiform Fibers: Nonoccupational Health Risks

Perkins and Harvey (1993) *Test Method: Method for the Determination of Asbestos in Bulk Building Materials*, EPA/600/R-93/116

Yamate et al. (1984) Methodology for the measurement of airborne asbestos by electron microscopy, U.S. Environmental Protection Agency Report

EXHIBIT A

CURRICULUM VITAE MELINDA DARBY DYAR

Department of Astronomy Mount Holyoke College South Hadley, MA 01075 (413) 538-3073 Planetary Science Institute 1700 East Fort Lowell, Suite 106 Tucson, AZ 85719-2395 520-622-6300 161 Chestnut St. Amherst, MA 01002 (413) 348-9424 mdyar@mtholyoke.edu

Education:

<u>Ph.D.</u>, <u>Geochemistry</u>, Massachusetts Institute of Technology, Cambridge, Mass. Thesis topic: Crystal chemistry and statistical analysis of iron in mineral standards, micas, and glasses. Advisor: Roger G. Burns.

<u>B.A., Geology and Art History</u>¹, Wellesley College, Wellesley, Mass. Thesis topic: Geology of the Broadmoor Wildlife Sanctuary, South Natick, MA: Structural and petrographic analysis. Advisor: Margaret Thompson. <u>Summer Field Camp</u>, Indiana University, Cardwell, Montana.

Employment:

Senior Scientist, Planetary Science Institute, 2015-present.

<u>Kennedy-Schelkunoff Professor and Chair</u>, Department of Astronomy, Mount Holyoke College and Five College Astronomy Department, 2011-present.

<u>Associate Professor and Chair</u>, Department of Astronomy, Mount Holyoke College and Five College Astronomy Department, 2002-2011.

<u>Associate Professor</u> Department of Earth and Environment, Mount Holyoke College and Five College Astronomy Department, 2002-2008.

Five College Graduate Faculty in Astronomy, University of Massachusetts, 2002-present.

<u>Visiting Associate Professor</u>, Department of Astronomy and Department of Earth and Environment, Mount Holyoke College and Five College Astronomy Department, 2001-2002.

<u>Visiting Associate Professor</u>, Department of Astronomy, University of Massachusetts (Amherst), 2001-2002.

Affiliated Staff, Department of Geological Sciences, University of Idaho, 2000-2018.

<u>Visiting Assistant Professor</u>, Department of Astronomy and Department of Earth and Environment, Mount Holyoke, 1998-2001.

<u>Assistant Professor</u>, Department of Geology and Astronomy, West Chester University, 1993-1998.

Visiting Assistant Professor, Department of Geology, Smith College, 1995-1996.

<u>Assistant Professor</u>, Department of Geological Sciences, University of Oregon, 1986-1993. Member, Materials Science Institute, 1987-1993.

<u>Research Fellow</u>, Division of Geological and Planetary Sciences, California Institute of Technology, 1985-1986 (G.R. Rossman, supervisor).

<u>Post-Doctoral Fellow</u>, Department of Earth, Atmospheric, and Planetary Sciences, Massachusetts Institute of Technology, 1985 (R.G. Burns, supervisor).

<u>Research Assistant</u>, Department of Earth, Atmospheric, and Planetary Sciences, Massachusetts Institute of Technology, 1980-1985.

Research Staff, Chevron Oil Field Research Company, La Habra, CA, summer, 1982.

Assistant Instructor, Indiana University Geologic Field Station, Cardwell, MO, summer, 1980.

Professional Societies:

Association for Women Geoscientists Geological Society of America (Fellow) Mineralogical Society of America (Fellow) American Geophysical Union North American Society for LIBS Geochemical Society (Fellow)

¹ Course requirements for the art history major were completed while a graduate student at MIT.

Honors:

Sigma Xi, 1980

1984 Mineralogical Society of America (M.S.A.) Grant for Research in Crystallography

1990-1991 National Lecturer, Mineralogical Society of America

Outstanding Service Award, Mineralogical Society of America, 1991

Fellow, Mineralogical Society of America, 1995

Girls' Incorporated, Holyoke, Massachusetts, Honoree, 1 April, 2004

Meribeth E. Cameron Faculty Award for Scholarship, Mount Holyoke College, 2010

Participating Scientist, Mars Science Laboratory Science Team, 2012-2014

J.K. Gilbert Award for outstanding contributions to planetary science, Geological Society of America, 2016

Fellow, Geological Society of America, 2017

Hawley Medal, 2017, Mineralogical Association of Canada

Eugene Shoemaker Distinguished Scientist Medal, NASA Solar System Exploration Research

Virtual Institutes, 2018

Helmholtz International Fellow, 2018-2021

Fellow, Geochemical Society, 2019.

Professional Service:

M.S.A. Research Grants Committee, 1991

M.S.A. Science Grants Committee, 1991

The Geochemical Society Program Committee, 1991-1994

National Science Foundation Instrumentation and Laboratory Instruction Panel, 1988 and 1992

National Science Foundation Undergraduate Curriculum and Course Development Panel, 1991

National Science Foundation Workshop: The Role of Faculty in the Disciplines in the

Undergraduate Education of Future Teachers, invited participant, contributor to working paper, 1992

Program Committee, Geological Society of America National Meetings, 1992-1994

American Geophysical Union Workshop: Shaping the Future of Undergraduate Earth Science Education, invited participant and contributor to working paper, 1996

M.S.A. Lecture Program Committee, 1998-2000; Chair 1999-2000

M.S.A. Crystallography Research Grant Committee 1998-1999

Council for Undergraduate Research (C.U.R.), Councillor, 1999-2000

American Geological Institute (A.G.I.) Outreach Committee, 2000-2003

Associate Editor, American Mineralogist, 2000-present

Review Panel, NASA Cosmochemistry Program, 2001

Review Panel, NSF/NATO Postdoctoral Fellowship Program, 2002-2003

Judges Panel, Name the Rovers contest, 2003

Review Panel, NSF, CCLI Program, 2003

Instrument Selection Review Panel, Mars Science Lander 2009, NASA, 2004

Review Panel, NSF, CCLI Program, 2004

International Program Committee, Goldschmidt Conference, 2005

Executive Committee, AWIS National Meeting, 2005

Review Panel, NASA, 2006

Review Panel, NASA, 2007

Review Panel, NASA, 2008

Review Panel, NASA, 2008

Review Panel, NSF, 2008

Review Panel, NASA, 2009

Review Panel, NSF, 2009

Review Panel, NASA, 2009

Review Panel, NASA, 2010

Review Panel, NASA, 2011

Review Panel, NASA, 2012

Review Panel, NASA, 2013

Review Panel, NASA, 2015

Review Panel, NASA, 2016

Review Panel, NSF, 2017 (×2)

Review Panel, NASA, 2018

Invited Talks and Professional Presentations:

University of California at Riverside, 1986 and 1991²

University of Maine at Orono, 1987 and 1991¹

Oregon State University, 1986 and 1991

Massachusetts Institute of Technology, 1987

Wellesley College, 1987

Smith College, 1988

State University of New York at Albany, 1989

Southern Methodist University, 1989

University of Washington, 1990

University of Saskatchewan, 1991¹

San Diego State University, 1991¹

University of California at Davis, 1991¹

Sonoma State University, 1991¹

University of Calgary, 1991¹

University of New Mexico, 1991¹

University of Houston, 1991¹

Louisiana State University, 1991¹

Texas Tech, 1991¹

University of Chicago, 1992

Penn State University, 1992

University of Kentucky, 1992

University of Colorado at Boulder, 1993

European Research Conference, Hydrogen-Containing Defects in Minerals and Ceramics, 1993

Deep Continental Studies Workshop, Microknowledge and Megathinking, 1993

Portland State University, 1993

Hopi Buttes Workshop, invited participant, 1993

Rutgers University, 1994

University of North Carolina, 1994

Southern Methodist University, 1994

University of Massachusetts at Amherst, 1996

Mount Holyoke College, 1997

University of Western Ontario, 2000

University of Idaho, 2001

Eastern Washington University, 2001

Wellesley College, 2001

State University of New York at Stony Brook, 2003

Lunar and Planetary Institute, 2003

Brearley School, 2004

²talk sponsored by Mineralogical Society of America Lecture Program.

Massachusetts Institute of Technology, 2004

University of Massachusetts, 2004

Wellesley College, 2004

Rhode Island College, 2006

SUNY Binghamton, 2007

University of New Mexico, 2007

Colby College, 2008

Rutgers University, 2009

NASA Summer Science Institute, Goddard Space Flight Center, 2009

Hartwick College, 2010

M.I.T., 2011

Wellesley College, 2011

Brown University, 2011

Indiana University, 2011

TED talk, 2012

Texas Tech University, 2013

Skidmore College, 2014

University of Massachusetts Amherst, 2014

Rutgers University, 2014

Planetary Science Institute, 2015

TED-X, Springfield, MA 2015

University of Massachusetts, Amherst, 2015

Brown University, 2015

Amherst College, 2016

Rensselaer Polytechnical Institute, 2017

M.I.T., 2017

Williams College, 2018

Keynote Plenary Speaker, AAAS, January 2018

Colby College, 2018

University of Vermont (Burlington), 2018

Goddard Space Flight Center, 2019

Applied Physics Laboratory, 2019

College and University Service:

Structural Geologist Search Committee, University of Oregon (UO) 1987-1988

Chair, Affirmative Action Search Committee, UO, 1988

Chair, X-ray and Thin Section Committee, Dept. of Geological Sciences, UO, 1986-1987

Faculty Advisor, Condon Undergraduate Society, UO, 1986-1988

Affirmative Action Liaison, Dept. of Geological Sciences, UO, 1986-1989

Materials Science Institute Budget Committee, UO, 1987-1992

Treasurer, Materials Science Institute, UO, 1988-1992

Director, Materials Science Institute Research for Undergraduates Program, UO, 1988 & 1989

Physics Department Condensed Matter Search Committee, UO, 1988 and 1989

Graduate Teaching Fellow Coordinator, Dept. of Geological Sciences, UO, 1988-1989

Seismology Search Committee, Dept. of Geological Sciences, UO, 1988-1989

Volcanologist Search Committee, Dept. of Geological Sciences, UO, 1989-1990, 1990-1991

Telephone Counselor, Dept. of Geological Sciences, UO, 1990-1991

Chair, Displays Committee for Cascade Hall, UO, 1990-1993

Computer Coordinator, Dept. of Geological Sciences, UO, 1989-1993

Member, President's Task Force on Campus Infrastructure and Technology, UO, 1990-1992

Member, Committee on Campus Hazardous Waste Remediation, UO, 1988-1993

Case 3:16-md-02738-MAS-RLS Document 9903-1 Filed 05/30/19 Page 83 of 137 PageID: 81372

Member, University Library Committee, UO, 1987-1989

Member, Faculty Advisory to the Museum of Art, UO, 1989-1993

Chair, Faculty Advisory to the Museum of Art, UO, 1990-1993

Member, Departmental Computer Lab Managers, UO, 1989-1993

Board Member, Faculty Club of the University of Oregon, 1988-1993

Secretary-Treasurer, Faculty Club of the University of Oregon, 1989-1990

Member, Docent Council, University of Oregon Museum of Art, 1988-1993

Member, Board of Governors, University of Oregon Museum of Art, 1988-1993

Member, Curriculum Committee, Dept. of Geology and Astronomy, WCU, 1993-1996

Member, College of Arts and Sciences Recruitment Committee, WCU, 1994-1995Internship

Coordinator, Dept. of Geology & Astronomy, WCU, 1993-1997³

Member, Undergraduate Review Committee, Dept. of Geology & Astronomy, WCU, 1993-1998²

Member, Graduate Review Committee, Dept. of Geology & Astronomy, WCU, 1994-1998²

Member, Fellowship Committee, Mount Holyoke College, 2002-2006

Member, Planning and Budget Committee, 2003-2004

Chair, Fellowship Committee, Mount Holyoke College, 2003-2006

Coordinator of Universal Application Funding, 2003-2012

Member, Search Committee for Fellowships and Pre-Graduate Advising, 2009-2010

Task Force on Curriculum to Career, 2011

Member, Curriculum Committee, Five College Astronomy Department, 1999-present

Member, Radiation Safety Committee, Mount Holyoke College, 1999-present

Chair, Astronomy Department, 1999-present

Senate member, Five College Astronomy Department, 1999-present

Advisory Committee, 2017-2018

Miscellaneous Presentations to the College Community and Beyond:

Mathematics Department, MHC, 2000

Five College Geology Symposium, February 2001

Mount Holyoke Club of Bridgeport, CT, 2003

Lyon Lecture series, Denver, 25 Oct. 2003

Family Weekend, 31 Oct. 2003

Mathematics Department, MHC, February 2004

Five College Geology Symposium, February 2004

Mars lecture, AST 11, Amherst College, 5 March 2004

Fascinating Professor program, Francis Perkins scholars, 9 March, 2004

South Hadley Lions Club, 6 April 2004

East Longmeadow Library, 1 May, 2004

Dedication of Kendade Hall, 8 May, 2004

Class of 1959 reunion dinner, 28 May 2004

Mount Holyoke Club of South Hadley, 9 June, 2004

NOVA/WGBY fundraiser, Mount Holyoke College, 14 June, 2004

Summer math for high school students, summer Mars project, July, 2004

MHC Office of Admissions, summer staff training seminar, August, 2004

Family Weekend, 30 Oct. 2004

Hughes Symposium on Integrating Undergraduates into Research Programs, January 2005

Mount Holyoke Club of Houston, March, 2005

Springfield Star Club, April, 2005

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³Maternity leave from West Chester University was taken during the academic years 1995-1996 and 1997-1998.

Mount Holyoke Reunion, June 2005

Century Club, Springfield, MA, March 2006

Mount Holyoke Reunion, June 2006

Mid-Coast Maine Mount Holyoke Club, June, 2006

MacDuffie School, January 2007

Mount Holyoke Club of New York City, March, 2007

Stargazers Club, North Scituate, RI, April 2007

Mount Holyoke Gala, Washington D.C., April 2009

Mount Holyoke Club of Bridgeport, December 2011

Development Office function in New York City, May 2012

Development Office function in San Francisco, October 2012

Development Office function in Los Angeles, October 2012

Development Office function in Boston, November 2012

Mount Holyoke Club of Cape Cod, May 2013

Mount Holyoke Club of New Hampshire, June 2013

Albany Area Amateur Astroomers, October, 2014

New England Society of Economic Geologists, January, 2015

Mount Holyoke Club of New Hampshire, October, 2015

Mount Holyoke Club of Fairfield County, October, 2015

Talk for 6th grade class at Common School, Amherst MA, May, 2017

Talk for the Development Office, October 2017

Reunion talk, class of 1967, May 2018

Courses Taught:

Mineralogy I and II (advanced undergraduate)

Igneous and Metamorphic Petrology (advanced undergraduate)

Introduction to Earth History (introductory historical geology)

Introduction to Geology (introductory physical geology)

Planet Earth (introductory geology/planetary science)

Geochemistry (graduate and undergraduate)

Spectroscopy (graduate and undergraduate)

Geometrics (applications of statistical methods) (graduate and undergraduate)

Planetary Science (introductory and advanced undergraduate)

Planetary Science seminar on Mars (advanced undergraduate)

Spectroscopy of the Planets (advanced undergraduate)

Meteorites (advanced undergraduate)

Books and Electronic Media:

Dyar, M.D., and Gunter, M.E. (2013) Mineral Database. App for I-pad, Mac, I-Phone.

McKillip, S. and Dyar, M.D. (2010) *Geostatistics Explained, An Introductory Guide for Earth Scientists*. Textbook. Cambridge University Press.

Dyar, M.D., and Gunter, M.E. (2008) *Mineralogy and Optical Mineralogy: A Three-Dimensional Approach*. Textbook. Mineralogical Society of America.

Dyar, M.D. (1999) *Hands-On Mineral Identification*. CD-ROM. Tasa Graphic Arts, Inc., Albuquerque, N.M.

Dyar, M.D., Busch, R.M., and Wiswall, G. (1997, 1998) *The Study of Minerals*. CD-ROM. Tasa Graphic Arts, Inc., Albuquerque, N.M.

Dyar, M.D., McCammon, C.A., and Schaefer, M.W., Eds. (1996) *Mineral Spectroscopy: A Tribute to Roger G. Burns*. Special Publication #5, The Geochemical Society, Washington, D.C., 400 pp.

Papers:

- 1. Dyar, M.D., and Burns, R.G. (1981) Coordination chemistry of iron in glasses contributing to remote-sensed spectra of the moon. *Proc. Lunar and Planet. Sci. Conf.*, **12B**, 695-702.
- 2. Burns, R.G., and Dyar, M.D. (1983) Spectral chemistry of green glass-bearing 15426 regolith. Proc. Lunar and Planet. Sci. Conf., 14, *J. Geophys. Res.*, **88**, B221-B228.
- 3. Dyar, M.D., and Birnie, D.P. (1984) The effects of quench media on iron partitioning and ordering in a lunar glass. Proc. 1st Intl. Conf. on Glass in Planet. and Geolog. Phenomena, *J. Non-Cryst. Sol.*, **67**, 397-412.
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- 528. Kashyap, S., Sklute, E., Ross, L., Emerson, D., Tague, T.J. Jr., Wang, P., Holden, J.F., and Dyar, M.D. (2018) Spectroscopic characterization of natural biogenic iron (oxyhydr)oxides from freshwater and marine environments. *Lunar Planet. Sci. XLIX*, Lunar Planet. Inst., Houston, (abstr.) #1130.
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- 556. Roberts, S.E., Sheffer, A.A., McCanta, M.C., Dyar, M.D., and Sklute, E.C (2019) Investigating redox change during impacts. *LPSC L*, Abstract #1285.

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Theses Supervised:

- Reich, D.R. (M.S., University of Oregon, 1989) Geology and Petrology of the Mt. Emily Volcanic Center.
- Hull, C.D. (Ph.D., University of Oregon, 1990) Multicomponent Chemical Equilibrium Modeling of Fluids and U-Th Geochronology of Minerals in Geothermal Systems.
- Harrell, M.D. (Honors College, B.A., University of Oregon, 1991) *In situ* Alteration of a Rock from the Earth's Mantle
- Moeller, K. J. (M.S., University of Oregon, 1991) Crystal Chemistry of Chlorites from Metapelites.
- Grant, C. G. (Ph.D., 1995, Chemistry, University of Oregon) Sources of Experimental and Analytical Error in Measurements of the Mössbauer Effect in Amphibole.
- Taylor, M.E. (M.A., West Chester University, 1996) Crystal Chemistry of Iron in Tourmaline.
- Voci, Christopher (M.A., West Chester University, 1996) Pb Stability in Pyromorphite from Landfills.
- Stefanis, M. (B.A., Mount Holyoke College, 1999) An Interpretation of the Rocks at the Mars Pathfinder Landing Site.
- Polyak, D.E. (B.A., Mount Holyoke College, 1999) Crystal Chemistry of Iron in Plagioclase from Four Heavenly Bodies.
- Blackwell, M.A. (B.A., Mount Holyoke College, 2000) Recoil-free Fraction Effects in Amphibole.
- Lowe, E.W. (B.A., Mount Holyoke College, 2000) Distribution Coefficients for Fe³⁺ and Fe²⁺ in Metapelites from Western Maine.
- Cartwright, B.M. (M.S., University of Massachusetts, 2003) Plagioclase Fe³⁺/Fe²⁺ Correlation with Magma Oxygen Fugacity in a Volcanic Succession: The Atascosa Mountains, South-central Arizona. Co-supervised with Sheila Seaman.
- Makuch, L.B. (B.A., University of Massachusetts, 2002) The Ares Project. Honors College.
- Therkelsen, J.B. (B.A., Amherst College, 2002) Shock-induced changes in redox state of experimentally shocked plagioclase, pyroxene, and olivine.
- Peek, K.M. (B.A., Mount Holyoke College, 2002) The disruption of an icy satellite and the evolution of the resulting debris ring. A formation scenario for Saturn's rings.
- Dickson, J. (B.A., Hampshire College, 2002) Water on Mars: A synthesis of hydrologic features on the surface of Mars.
- Daane, A.R. (B.A., Mount Holyoke College, 2004) Brightness and color variations in the hot pulsating horizontal branch star PG1627+017.
- Hunter, M.O. (B.A., Mount Holyoke College, 2004) Pacific Equatorial Current systems during the waning of the 2002-2003 El Niño.

- O'Connor, V. (B.A., Smith College, 2005) Comparative crystal chemistry of hydrous iron sulfates from different terrestrial environments.
- Dopfel, E. (B.A., Mount Holyoke College, 2006) The chemical activators of cathodoluminescence in jadeite.
- Sklute, E.C. (B.A., Mount Holyoke College, 2006) Mössbauer spectroscopy of synthetic olivine across the Fe-Mg solid solution.
- Rothstein, Y. (B.A., Mount Holyoke College, 2006) Spectroscopy of jarosite and implications for the mineralogy of Mars.
- Howenstine, J. (B.A., University of Massachusetts, 2006) Analysis of depth-diameter relationship of martian craters.
- Bendersky, C. (B.A., Mount Holyoke College, 2007) The onset of thermal metamorphism in enstatite chondrites.
- Walker, C.A. (B.A., Mount Holyoke College, 2007) Variations of solar wind parameters over a solar cycle: Expectations for NASA's Solar Terrestrial Relations Observatory (STEREO) Mission.
- Graham, S. (B.A., Mount Holyoke College, 2007) GPS geodetic constraints on the November 21, 2004 Mw 6.3 earthquake off the northwest coast of Dominica: implications for in situ volatile solubilities and eruptions dynamics.
- Barkley, M. (B.A., Mount Holyoke College, 2007) The effects of F-OH⁻ substitution on the crystal structure of pegmatitic topaz.
- Cadieux, Sarah Beth (B.A., Mount Holyoke College, 2008) Carbon-isotope stratigraphy of Upper Cretaceous deposits of the Dalmatian Island of Brač, Croatia.
- Langford, Amy (B.A., Mount Holyoke College, 2008) Unbiased search for molecular outflows in the Taurus Molecular Cloud.
- Emerson, Erica (B.A., Mount Holyoke College, 2008) Analysis of iron oxidation in garnets.
- Tucker, Jonathan (B.A., Amherst College, 2009) Calibrating ChemCam: Preparing to Probe the Red Planet.
- Knutson, J.K. (B.A., Mount Holyoke College, 2010) Exploring biotite iron transformation by the hyperthermophilic archaeon Pyrobaculum Islandicum.
- Barbieri, Lindsay (B.A., Hampshire College, 2010) Deciphering Late-Amazonian climate change on Mars using evidence preserved in gully fan stratigraphy.
- Bell, S. (B.A., Amherst College, 2011) Fresh lunar crater ejecta as revealed by the Miniature radio Frequency (Mini-RF) instrument on the Lunar Reconnaissance Orbiter.
- Ozanne, M.V. (B.A. Mount Holyoke College, 2012) Comparison of shrunken regression methods for major elemental analysis of rocks using laser-induced breakdown spectroscopy (LIBS).
- DeVeaux, M.L. (B.A. Mount Holyoke College, 2012) Evaluation of statistical methods for classification of laser-induced breakdown spectroscopy (LIBS) data.
- Williams, Molly (B.A., Mount Holyoke College, 2012) Characterizing Fe(III) transformation in a deep-sea hyperthermophilic archaeon.
- Kashyap, Srishti (B.A., Mount Holyoke College, 2013) Isolating and characterizing an Fe(III) transforming deep sea thermophilic archaeon.
- Breitenfeld, Laura (B.A., Mount Holyoke College, 2017) Predicting olivine composition using Raman spectroscopy through band shift and multivariate nnalyses.
- Ytsma, Caroline (B.A. Smith College, 2018) Univariate and multivariate quantification of hydrogen, lithium, boron, carbon, and sulfur under vacuum and in Earth and Mars atmospheres using laser-induced breakdown spectroscopy.
- Carey, CJ (Ph.D., University of Massachusetts, 2018) Graph construction for manifold discovery.
- Boucher, Thomas (Ph.D., University of Massachusetts, 2018) Transfer learning with mixtures of manifolds.

Graduate Committees:

McCanta, Molly (Ph.D., Brown University, 2004)

Buczkowski, Debra (Ph.D., University of Massachusetts, 2006)

Keskula-Snyder, Anna (Ph.D., University of Massachusetts, 2006)

Klima, Rachel (Ph.D., Brown University, 2007)

Amy Stander (MS, URI, 2013)

Tommy Boucher (Ph.D., UMass, 2018 Computer Science)

C.J. Carey (Ph.D., UMass, 2017, Computer Science)

S. Giguere (Ph.D., UMass, current student, Computer Science)

S. Kashyap (Ph.D., UMass, current student, Microbiology)

EXTERNAL GRANT:

PREVIOUS FUNDING:

American Chemical Society, Petroleum Research Fund:

"Cation Ordering in Synthetic Trioctahedral Micas"

\$18,000 funded, 7/1/87-8/31/89

\$4,500 supplement, 5/1/88-9/30/88

\$2,500 supplement, 5/1/89-7/1/89

"Detailed Crystal Chemistry of Iron-Bearing Phyllosilicates"

\$40,000 funded, 5/1/91-8/31/93

\$3,000 supplement, 6/1/91-9/30/91

National Science Foundation, Earth Sciences Division:

"Crystal Chemistry and Petrogenesis of Biotites from Pelitic Schists"

\$60,000 funded, 7/1/87-6/30/90

"Crystal Chemistry of Metapelitic Minerals in a Petrologic Context"

\$50,226 funded, 1/1/89-12/31/90

"Crystal Chemistry of Hydrogen and Oxygen in Common Phyllosilicate Minerals"

\$61,774 funded, 1/1/92-12/31/93

"Development of Standards for Light Element Analysis in Geological Materials: Collaborative Research" \$ 21,639 funded, 6/1/93-12/31/95

"The Boron Budget in High-Grade Pelitic Metamorphic Rocks: How, When, and Where does the Boron Go?" (Joint with C.V. Guidotti and E.S. Grew)

\$56,333 funded for period 1/1/96-12/31/97

"Hydrogen Partitioning and Fe³⁺ Exchange in Mantle Minerals: Effects on Mechanical Behavior -

Collaborative Research" (Joint with S.J. Mackwell)

\$142,997 funded for period 11/1/93 to 5/31/98

\$5,000 supplement funded for 5/1/94 to 9/1/94

"Redox Processes in Rio Grande Rift and Colorado Plateau Xenoliths: Collaborative Research"

(Joint with A.V. McGuire)

\$55,598 funded for period 6/1/94-5/31/98

\$7,000 supplement funded for 5/1/95 to 9/1/95

"Synchrotron MicroXANES Study of Iron Redox in Mantle Phases"

\$60,000 funded for period 8/15/98 - 2/28/01

"R.U.I.: Acquisition of a New Mössbauer spectrometer"

\$49,840 funded for period from $3/\overline{1/99}$ - 2/28/01

"R.U.I.: Collaborative Research: Chemical Equilibria Involving Iron and Hydrogen in Metapelites from Western Maine"

\$27,468 funded for period from 8/1/99 - 7/31/01

"RUI: Acquisition of a Scanning Electron Microscope at Mount Holyoke College"

\$226,560 funded for period from 6/1/02-5/30/03

"RUI: Collaborative Research: Hydrogen and Ferric Iron in Felsic Melts"

(collaborative with Sheila Seaman, University of Massachusetts)

\$86,000 funded for period from 1/1/03-12/31/06

"RUI: Collaborative Research: Improvements in the Application of the Mössbauer Effect to Studies of Minerals"

(collaborative with Martha Schaefer, LSU)

\$76,939 funded for period from 1/1/05-12/31/08

"Redox Ratios by Fe-XANES"

(collaborative with M. Gunter and A. Lanzirotti)

\$104,897 funded for period from 6/1/08-5/31/11

"Effects of Composition and Cooling Rate on Fe XANES Glass Calibrations"

(Collaborative with Molly McCanta at Tufts University)

\$115,363 funded to MHC for period from 8/1/12 - 7/31/15

National Science Foundation, Division of Computation and Data-Enabled Science and Engineering:

"Transfer Learning for Chemical Analyses from Laser-Induced Breakdown Spectroscopy"

(Sridhar Mahadevan, UMass, Co-I)

\$141,129 to MHC for period from 9/1/13 - 8/31/15

National Science Foundation, Division of Undergraduate Education:

"Undergraduate Research Program in Materials Science"

\$14,500 funded, 5/1/88-9/30/88

"Undergraduate Research Program in Materials Science"

\$36,600 funded, 5/1/89-12/31/89

"Equipment for Program Improvement in Mineralogy/Petrology"

\$45,000 funded, 1/1/89-12/31/89

\$40,000 match from University of Oregon

\$7,500 match from I.B.M. and Novell

"Support for Development of Minerals and Mineralogy: A Three-Dimensional Approach"

\$75,000 funded for period from 3/1/00 - 2/28/02

"A Modular, Inquiry-Based Regional Approach to Introductory Laboratories"

(S. Good, C.G. Fisher, R.M. Busch, T.M. Lutz, L. Srogi, and C.G. Wiswall, Co-I's) NSF grant DUE-9850923, \$40,800 funded, 6/1/98-5/31/2000.

"An Integrative Curriculum in Earth, Human, and Environmental Sciences

(Joint with L. Savoy, M.J. Markley, A. Werner, M.A. McMenamin, S. Dunn, and T. Millete)

\$170,000 funded for period from 1/1/00 - 6/30/05

"Support for Development of Minerals and Mineralogy: A Three-Dimensional Approach"

\$417,244 funded for period from 3/1/02 - 2/28/08

"An Integrative Curriculum in Planetary Science"

(C.M. Hamilton, T. Burbine, co-Is)

\$91,900 funded for period from 9/1/05 - 8/31/07

"Scaffolding Effective Practice for Use of Animations in Teaching Mineralogy and Physical Geology"

(M. Gunter and L. Wenk, Co-I's)

\$150,000 funded for period from 1/1/09 -12/31/10

"Building Analytical Competence for Geoscience Students through use of Spectroscopic Tools"

(Collaborative with John B. Brady and Eileen McGowan)

\$73,375 funded to MHC for period 8/15/12-7/31/15

National Science Foundation Scholarships in Science, Technology, Engineering, and Mathematics

"Improving Recruitment and Retention of Transfer Students to STEM Majors at Mount Holyoke College" (Sarah Bacon and Becky Packard, Co-Is)

\$600,000 for period from 1/1/12-12/31/16

Packard Foundation

"Earth and Environmental Sciences in California and the Southwest" (Joint with A. Ellison, L. Savoy, and T. Millette)

\$200,000 funded for period from 1/1/00 - 12/31/02

Tektronix Foundation:

"Request for Upgrade to Mössbauer Spectroscopy Laboratory" \$4,000, 1987; \$6,000, 1988; \$10,000, 1989

NASA, Cosmochemistry Division:

"Synchrotron microFTIR characterization of hydrogen in nominally anhydrous minerals in martian materials."

\$68,000 funded for period from 1/1/01 - 12/31/02

"Ferric Iron and Hydrogen in Mars Minerals"

\$45,000 funded for period from 1/1/03 - 12/31/03

"Acquisition of a 4.5K Mössbauer Spectrometer"

\$35,345 funded for period from 1/1/05 to 12/31/06

NASA, Exobiology Program:

"Biogenic iron oxide transformations by thermophilic and mesophilic iron-reducing microbes" James Holden, University of Massachusetts, PI \$208,316 to MHC for period from 4/1/14-3/31/18

NASA, Mars Fundamental Research Program:

"Temperature Dependence and Resolution of Fundamental Mössbauer Parameters in Mars-Analog Minerals"

\$150,000 funded for period from 10/1/02 - 9/30/06

"Taking Apart the Rocks of Mars" (C. Pieters, P.I., J. Bishop, J. Sunshine, T. Hiroi, Co-Is) NNG04GB53G

\$60,000 funded for period from 12/31/03 - 11/30/06

"Analysis and Characterization of Sulfates and Sulfides Using Multiple Spectral Techniques (M. Lane, P.I., J.L. Bishop, Co-I)

\$31,000 funded for period from 1/1/05-12/31/06

"Temperature Dependence and Resolution of Fundamental Mössbauer Parameters in Mars-Analog Minerals"

(collaborative with M. Schaefer, LSU)

\$90,000 funded for period from 3/1/06-2/28/09

"Mineral Standards and Technique Development for Laser-Induced Breakdown Spectroscopy" \$375,000 funded for period from 3/1/06-2/28/09

"Formation of Magnetic Minerals on Mars by Alteration of Nanophase Ferric Oxides/Oxyhydroxides" (Janice Bishop, SETI/Ames, PI)

\$30,000 funded for period from 3/1/06-2/28/09

"Further Analysis and Characterization of Sulfates and Sulfides Using Multiple Spectral Techniques" (collaborative with Melissa Lane, Planetary Science Institute (PI), and Janice Bishop, SETI) \$32,000 funded for period from 3/1/06-2/28/09

"Characterizing the Rocks of Mars through Integrated Spectroscopy"

(collaborative with C. Pieters (PI), J. Bishop, M. Lane, T. Hiroi, J. Sunshine)

60,000 to MHC for period from 3/1/07 - 2/28/11

"Analysis and Characterization of Phosphates Using Multiple Spectral Techniques"

(collaborative with Melissa Lane (PI), Planetary Science Institute, and Janice Bishop, SETI) \$45,947 funded for period from 3/1/08-2/28/12

"The State of Sulfur on Mars: Understanding the Interrelationships Among the Crystal Structure,

Chemistry, and Spectroscopy of Sulfates and Sulfides "

(collaborative with Melissa Lane (PI), Planetary Science Institute, and Janice Bishop, SETI)

\$97,754 funded to MHC for period from 6/1/10-5/31/13

"Technique Development for Laser-Induced Breakdown Spectroscopy: Calibration, Classification, and Light Element Analysis"

(collaborative with Sam Clegg and Roger Wiens, LANL, and Martha Schaefer)

\$719,000 funded for period from 3/1/09 - 5/30/14

"Effects of Shock Metamorphism on Phyllosilicate Spectroscopy"

(Joseph Michalski, P.I.)

\$67,941 funded to MHC for period from 6/1/10-5/31/14

"Impact History and Meteoritic Contributions to the Martian Upper Crust"

(collaborative with Caleb Fassett, PI)

\$261,888 funded to MHC for period 6/1/14-5/31/18

"Transfer learning for chemical analyses from laser-induced breakdown spectroscopy on the surface of Mars"

(collaborative with Sridhar Mahadavean)

\$445,093 funded to MHC for period 11/29-14-11/30/17

NASA, Lunar Advanced Science and Exploration Research Program:

"Thermodynamics, VIS-IR Spectroscopy, and Mapping of Adsorbed Water on the Moon"

(K. Hibbitts, PI)

\$33,920 to MHC for period from 6/1/08 - 5/31/09

"Pyroxene Spectroscopy as a Tool to Probe the Composition and Thermal History of the Lunar Surface" (Rachel Klima, PI)

104,914 to MHC for period from 1/1/10 - 5/31/13

"The Stability and Transport of Water on the Moon"

(Karl Hibbitts, P.I.)

\$106,614 funded to MHC for period from 9/1/11-6/30/15

"Solar Wind Induced Production of Hydroxyl and Water on the Moon"

(Thomas Orlando, PI)

\$9,313 to MHC for period from 9/1/11-6/30/15

NASA, Mars Science Laboratory Participating Scientist Program:

"Enhancing Science Return from ChemCam through Laboratory and Statistical Analyses and Integration with APXS"

\$476,885 for period from 2/15/12-12/31/16

NASA, Lunar Science Institute Program:

"The Moon as Cornerstone to the Terrestrial Planets: The Formative Years"

C.M. Pieters, PI, Brown University

\$135,000 funded to MHC for period from 4/9/09 - 4/8/14

NASA Planetary Geology and Geophysics Program:

"Integrated Spectroscopy of Pyroxenes: Composition, Structure, and Thermal History" (Rachel Klima, PI)

\$32,711 to MHC for period from 8/5/11-8/4/14

"Pyroxene Spectroscopy, Composition, Structure, and Thermal History" (Rachel Klima, PI)

\$91,313 to MHC for period from 6/1/13 - 5/31/16

NASA Solar System Workings Program:

"SuperCam-Analog Laser-induced Breakdown Spectrometer at Mount Holyoke College" \$445,095 for period 10/1/16-9/30/18

CURRENT FUNDING:

NASA, Solar System Exploration and Research Virtual Institute Program:

"Remote, In Situ, and Synchrotron Studies for Science and Exploration"

Tim Glotch, Stony Brook Univ., PI (Dyar is Deputy PI)

\$777,256 to MHC for period from 1/1/14-12/31/18

"Volatiles, Regolith and Thermal Investigations Consortium for Exploration and Science (VORTICES)"
Ben Bussey, APL, PI

\$51,998 to PSI for period from 1/1/14-12/31/18

"Environment and Evolution of Exploration Destinations: Science and Engineering Synergism"

Carle Pieters, Brown Univ., PI

\$135,600 to MHC for period from 1/1/14-12/31/18

"TREX: Toolbox for Research and Exploration"

Amanda Hendrix, PSI, PI

5,483,517 to PSI 6/1/17 - 5/31/22

NASA, Exobiology Program:

"ICE-MAMBA Ice-covered Chemosynthetic Ecosystems: Mineral Availability and MicroBiological Accessibility"

Jill Mikuchi, Univ. of Tennessee, PI

\$208,316 to PSI for period from 1/22/17-1/21/20

"Fe(III) Oxide Reduction by a Hyperthermophillic Crenaraeon: Novel Mechanisms and Detection"

James Holden, Univ. of Massachusetts, Amherst, PI

\$230, 236 to Planetary Science Institute for period from 10/1/18 - 9/30/21

NASA Emerging Worlds Program:

"Volatile Adsorption onto Primitive Grains for Understanding the Formation of the Early Solar System" Karl Hibbitts, PI

\$13,858 to PSI for period from 3/1/5-2/28/18

NASA Solar System Workings Program:

"Oxidation State of Lunar Glasses: Implications for Surface and Eruptive Processes on the Moon"

Molly McCanta, PI

\$136,530 to PSI for period from 10/1/16-3/30/19

"Towards Quantifying Oxygen Fugacity in Solar System Materials: In Situ Multivalent Element Analyses in Pyroxene"

Molly McCanta, PI

267,184 to Dyar for period from 10/1/18 - 9/30/21

NASA Planetary Data Archiving, Restoration, and Tools:

"Advanced XAS Calibration Tools for In Situ Analysis of Redox States of Fe, Ti, Mn, Cr, V, and Eu in Extraterrestrial Glasses"

\$208,997 to PSI for period from 1/15/17-1/14/20

NSF Information Integration and Informatics

"Collaborative Research: Deep Learning for Spectroscopy" Collaborative with Sridhar Mahadevan and Mario Parente \$373,793 to MHC for period from 4/1/16-3/31/20

NSF Petrology and Geochemistry:

"Refining Geothermobarometry in Pyroxenes using In Situ Measurements of Fe³⁺" \$172,000 fto MHC or period from 7/1/18 - 6/30/2101

NSF Planetary Astronomy:

"Formation, Stability, and Detection of Amorphous Ferric Sulfate Salts on Mars" Elizabeth Sklute, PI \$232,273 to MHC for period from 10/1/18 – 9/30/21

EXHIBIT B

Previous Four Years of Expert Testimony for Melinda Darby Dyar, Ph.D.

Dr. Melinda Darby Dyar has not testified as an expert at trial or by deposition during the previous four years.